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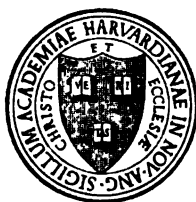


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JOURNAL

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS

BOARD OF EDITORS

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L. L. VAN SLYKE

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No. 1

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PART II

Report of Committee on Editing Methods of Analysis
Leathers
Insecticides and Fungicides
Foods and Feeding Stuffs
Saccharine Products
Food Preservatives

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VI. LEATHERS.—TENTATIVE.

VEGETABLE TANNED LEATHER.

1

PREPARATION OF SAMPLE.

Grind the sample, without undue heating, and pass through a 10 mesh sieve. The ground sample must not contain hard lumps. Plane heavily greased leathers (containing more than 20 % fat) into very thin shavings. Spread out the prepared sample and allow it to return to atmospheric moisture condition; mix thoroughly, and place in tightly covered containers.

2

MOISTURE.

Place 10 grams of the sample, as prepared under 1, in a tared, wide, shallow, weighing bottle (or a similar dish which can be covered tightly), and dry in a water oven for 15 hours at 98°–100°C. Cover the weighing bottle, cool in a desiccator containing sulphuric acid, and weigh. The moisture present in the leather as received may be determined by cutting it quickly into small pieces and drying without grinding as directed above.

3

TOTAL ASH.

Incinerate slowly 5 grams of the sample, as prepared under 1, at a dull red heat. If difficulty is experienced in burning off the carbon, leach the residue with hot water, filter on an ashless filter, dry and ignite the filter and residue, add the filtrate, evaporate to dryness and ignite. Cool in a desiccator containing sulphuric acid and weigh.

The ash may be examined for acids and bases by any suitable method. Aluminium, magnesium, sodium, barium, calcium and lead are the bases, and hydrochloric and sulphuric acids are the acids which it may be necessary to determine.

4

INSOLUBLE ASH.

Incinerate slowly the residue from the extraction of water-soluble material, obtained in 6 or 7, until all the carbon is burned off, cool in a desiccator containing sulphuric acid and weigh.

5

FATS.

Place, without packing, 15 grams of the leather, as prepared under 1, in a Soxhlet or Johnson extractor with a layer of fat-free cotton above and below the sample. Extract 8–10 hours with petroleum ether distilling between 50° and 80°C. Heavily greased leathers (containing 15% or more fat) will require the maximum time. Remove the receiving flask, evaporate the petroleum ether on the steam bath and dry the fat residue for 3 hours in a water oven at 98°–100°C., cool in a desiccator and weigh. Repeat the drying in the water oven for periods of 1–1½ hours, cooling and weighing as before, until no further loss in weight occurs. Retain the leather residue from the fat extraction for the extraction of water-soluble material in 6 or 7.

EXTRACTION OF WATER-SOLUBLE MATERIAL.

6

Method I.

Evaporate the petroleum ether from the fat-free leather, obtained under 5, and moisten thoroughly with from 100–150 cc. of water. Place a layer of cotton in the

bottom of a Soxhlet extractor designed for making extractions at temperatures below 100°C.

An extractor of this kind is furnished with a water jacket surrounding that portion of the apparatus containing the sample but does not enclose the side tube which carries the hot vapors to the condenser.

Transfer the moistened fat-free leather to the extractor, and cover this with another layer of cotton to avoid siphoning off solid particles. Maintain the temperature of the jacket surrounding the Soxhlet at 50°C. (1) Pour 200 cc. of water (including that used in moistening the leather) into the Soxhlet and allow it to siphon into the flask below, then heat and extract for an hour. Remove the flame and transfer the extract to a liter graduated flask. Then add water and continue the extraction as directed below, removing and transferring the extract to the liter flask before each fresh addition of water.

(2) Add 175 cc. of water and extract for 2 hours.

(3) Add 175 cc. of water and extract for 3 hours.

(4) Add 175 cc. of water and extract for 4 hours.

(5) Add 175 cc. of water and extract for 4 hours.

Transfer the last portion of the extract to the graduated flask. This gives 14 hours' extraction and an extract which does not exceed 1 liter in volume. Dilute to 1 liter at room temperature and mix thoroughly.

7

Method II.

(This method is the same in principle as the official method of the American Leather Chemists Association.¹)

Digest overnight 30 grams of the fat-free leather, obtained under 5, in approximately 200 cc. of water. Transfer the leather and extract to a percolator. Continue the extraction by percolating with water at 50°C. Collect 2 liters of percolate, regulating the flow of water at such a rate that 2 liters will be collected in 3 hours. Dilute to volume at room temperature and mix thoroughly.

To the extract, prepared according to 6 or 7, add a few drops of toluol to prevent fermentation of sugars, and reserve for the determination of glucose, total solids, soluble solids, and nontannins.

GLUCOSE.

8

PREPARATION OF SOLUTION.

To 200 cc. of the leather extract, as prepared under 6 or 7, add 25 cc. of a saturated solution of normal lead acetate, mix thoroughly, and filter at once through a dry, plaited paper, returning the first portions of the filtrate to the filter until the filtrate becomes clear. Keep the containers and the funnel covered during these operations. Without waiting for the entire filtrate to run through add 10-12 grams of solid potassium oxalate, shake frequently during 15-20 minutes and filter through a dry, plaited paper returning the first runnings to the filter until the filtrate runs clear. Pipette 150 cc. of the last filtrate into a 600 cc. Erlenmeyer flask, add 5 cc. of concentrated hydrochloric acid and boil under a reflux condenser for 2 hours. Cool, neutralize with solid sodium carbonate, using a little phenolphthalein as indicator, transfer to a 200 cc. volumetric flask and complete to volume with water. Filter through a double filter, and return the first runnings until the filtrate becomes perfectly clear. Determine the dextrose in the filtrate immediately.

9

DETERMINATION.

Determine dextrose in 50 cc. of the solution, as prepared under 8, equivalent to 0.5 gram of leather, according to VIII, 25 and express the result as glucose.

10

TOTAL SOLIDS.

Determine as directed under V, 2.

11

SOLUBLE SOLIDS.

Determine as directed under V, 4.

12

NONTANNINS.

Determine as directed under V, 7.

13

SOLUBLE TANNIN.

The difference between the percentage of the soluble solids and the corrected nontannins is the percentage of tannin.

14

NITROGEN.

Determine as directed under I, 21.

15

HIDE SUBSTANCE.

Multiply the percentage of nitrogen by 5.62. The result will be the percentage of hide substance present.

16

COMBINED TANNIN.

Deduct the sum of the percentages of moisture, under 2, insoluble ash, under 4, soluble solids, under 11, and hide substance, under 15, from 100. The result will be the percentage of combined tannin.

BIBLIOGRAPHY.

¹ J. Am. Leather Chem. Assn., 1915, 10: 122.

VII. INSECTICIDES AND FUNGICIDES.

GENERAL METHOD.

1 PREPARATION OF SAMPLE.—TENTATIVE.

Mix thoroughly all samples before analysis. Make water-soluble arsenic determinations on samples as received without further pulverisation or drying. In the case of lye, sodium cyanid or potassium cyanid, weigh large quantities in weighing bottles and analyze aliquots of the aqueous solutions.

PARIS GREEN.

2 MOISTURE.—TENTATIVE.

Dry 2 grams at 105°–110°C. for 5 hours and express the loss in weight as moisture.

TOTAL ARSENIC.—OFFICIAL.

(Arsenic, present as arsenate, is titrated as arsenious oxid.)

3 REAGENTS.

(a) *Starch indicator*.—Mix about 0.5 gram of finely powdered potato starch with cold water to a thin paste; pour into about 100 cc. of boiling water.

(b) *Standard arsenious oxid solution*.—Dissolve 2 grams of pure arsenious oxid in a beaker by boiling with about 150–200 cc. of water containing 10 cc. of concentrated sulphuric acid, cool, transfer to a 500 cc. graduated flask and dilute to the mark.

(c) *Standard iodine solution*.—Prepare an approximately N/20 solution as follows: Mix intimately 6.35 grams of pure iodine with twice its weight of pure potassium iodid. Dissolve in a small amount of water, filter and dilute the filtrate to 1 liter in a liter graduated flask. Standardize against (b) as follows: Pipette 50 cc. of the arsenious oxid into an Erlenmeyer flask, dilute to about 400 cc., neutralize with sodium bicarbonate, add 4–5 grams in excess, and add the standard iodine solution from a burette, shaking the flask continuously, until the yellow color disappears slowly from the solution, then add 5 cc. of the starch indicator and continue adding the iodine solution, drop by drop, until a permanent blue color is obtained. Calculate the value of the standard iodine solution in terms of *arsenious oxid* (As_2O_3) and *arsenic oxid* (As_2O_5). Occasionally restandardize the iodine against freshly prepared arsenious oxid solution.

4 APPARATUS.

The apparatus used is shown in Fig. 5. The distillation flask rests on a metal gauze which fits over a circular hole in a heavy sheet of asbestos board. The first 2 Erlenmeyer flasks are of 500 and 1000 cc. capacity and contain about 40 and 100 cc. of water, respectively. Both of these flasks should be placed in a pan and kept surrounded with cracked ice and water. The third flask, containing a small amount of water, is used as a trap.

5

DETERMINATION.

Weigh an amount of the sample equal to the arsenious oxid equivalent of 250 cc. of the standard iodine solution, and wash into the distillation flask by means of 100 cc. of concentrated hydrochloric acid (sp. gr. 1.19). Add 5 grams of cuprous chlorid (Cu_2Cl_2) and distil.

When the volume in the distillation flask is reduced to about 40 cc., add 50 cc. of concentrated hydrochloric acid by means of the dropping funnel and continue the distillation until 200 cc. of the acid distillate have passed over. Then wash down the condenser and all the connecting tubes carefully, transfer these washings and the contents of the 3 Erlenmeyer flasks to a liter graduated flask and dilute to the mark. Mix thoroughly, pipette 400 cc. into an Erlenmeyer flask and nearly neutralize with a saturated solution of sodium or potassium hydroxid, using a few drops of phenolphthalein as an indicator, keeping the solution well cooled.

Continue as directed under 3 (C) beginning with "neutralize with sodium bicarbonate." The number of cc. of iodine used in this titration represents directly the total per cent of arsenic in the sample expressed as arsenious oxid (As_2O_3).

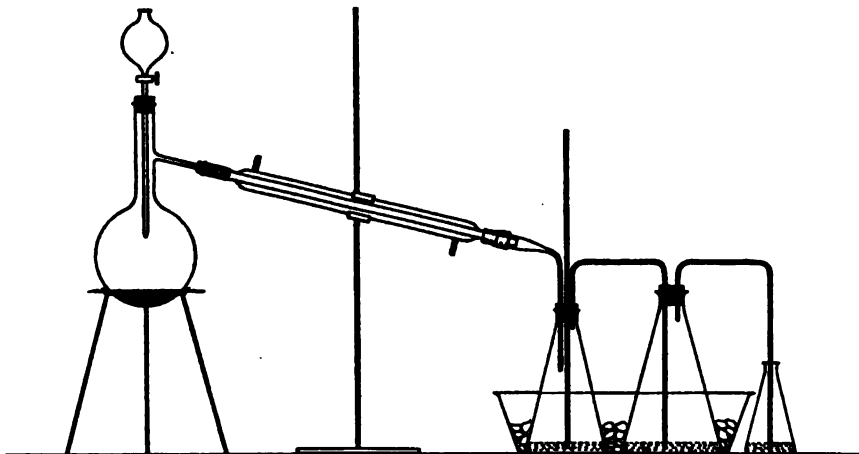


FIG. 5. APPARATUS FOR DISTILLATION OF ARSENIC CHLORID.

TOTAL ARSENIOUS OXID.

(The following methods determine arsenic, and antimony if present, as the -ous oxids, As_2O_3 and Sb_2O_3 , respectively. Ferrous and cuprous salts vitiate the results.)

*Method I.**C. C. Hedges Method,² Modified.³—Tentative.*

6

REAGENTS.

The reagents and solutions used are described under 3.

7

DETERMINATION.

Weigh an amount of the sample equal to the arsenious oxid equivalent of 100 cc. of the standard iodine solution, wash into an Erlenmeyer flask with 10–15 cc. of dilute hydrochloric acid (1 to 1), followed by about 100 cc. of water, and heat on the steam-bath to complete solution, at a temperature not exceeding 60°C . Cool, neutralize

with sodium bicarbonate, add 4-5 grams in excess, and then sufficient 25% ammonium chlorid solution to dissolve the precipitated copper. Dilute somewhat and titrate as directed under 3 (C). A correction must be applied for the amount of iodine solution necessary to produce a blue color with starch in the presence of copper (using an equivalent weight of copper sulphate). The corrected number of cc. of the standard iodine solution used represents directly the per cent of arsenious oxid (As_2O_3) in the sample.

Method II.

8 *C. M. Smith Method,³ Modified.—Tentative.*

Proceed as directed in 7, using dilute sulphuric acid (1 to 4) instead of dilute hydrochloric. The solution in this case may be heated to boiling.

SODIUM ACETATE-SOLUBLE ARSENIOS OXID.⁴—TENTATIVE.

9

REAGENTS.

(a) *Sodium acetate solution.*—Prepare a solution containing 12.5 grams of the crystallized salt ($CH_3COONa \cdot 3H_2O$) in each 25 cc.

The other reagents are described under 3.

10

DETERMINATION.

Place 1 gram of the sample in a 100 cc. flask and boil for 5 minutes with 25 cc. of the sodium acetate. Dilute to the mark, shake, and pass through a dry filter paper. Titrate an aliquot of this filtrate as directed under 3 (C). Calculate the amount of arsenious oxid (As_2O_3) present and express the result as per cent of sodium acetate-soluble arsenious oxid.

WATER-SOLUBLE ARSENIOS OXID.—TENTATIVE.

11

REAGENTS.

Described under 3.

12

DETERMINATION.

To 1 gram of the sample in a liter Florence flask add 1 liter of recently boiled water which has been cooled to exactly 32°C. Stopper the flask and place in a water bath kept at 32°C. by means of a thermostat. Digest for 24 hours, shaking hourly for 8 hours during this period. Filter through a dry filter and titrate 250 cc. of the filtrate as directed under 3 (C). Correct for the amount of the standard iodine necessary to produce the same color, using the same reagents and volume. Calculate the amount of arsenious oxid (As_2O_3) present and express the result as per cent of water-soluble arsenious oxid.

TOTAL COPPER OXID.

13

Electrolytic Method.—Official.

Treat 2 grams of the sample in a beaker with 100 cc. of water and about 2 grams of sodium hydroxid and boil thoroughly until all the copper is precipitated as cuprous oxid. Filter, wash well with hot water, dissolve the precipitate in hot dilute nitric acid, cool, transfer to a 250 cc. graduated flask and dilute to the mark. (1) Use 50-100 cc. of this solution for the electrolytic determination of copper as directed under VIII, 33 and calculate to per cent cupric oxid; or, (2) Electrolyze the aliquot in a weighed 150 cc. platinum dish, using a rotating spiral anode and a current of about

3 amperes. After all the copper is deposited (requiring about 30 minutes), wash the deposit with water by siphoning, then rinse with alcohol, dry for a few minutes in an oven, weigh and calculate to per cent cupric oxid.

14

*Thiosulphate Method.*⁴—Official.

Determine copper in another aliquot of the nitric acid solution of copper oxid, under 13, by titrating with N/20 thiosulphate solution, as directed under VIII, 29, and calculate to per cent cupric oxid.

LONDON PURPLE.

15

MOISTURE.—TENTATIVE.

Determined as directed under 2.

TOTAL ARSENIOS OXID.⁴—OFFICIAL.

16

REAGENTS.

Described under 3.

17

DETERMINATION.

Dissolve 2 grams of the sample in a mixture of about 80 cc. of water and 20 cc. of concentrated hydrochloric acid at a temperature of 60°–70°C.; filter and wash until the combined filtrate and washings measure 250 cc. Treat 100 cc. of this solution with sodium bicarbonate in excess, transfer to a 500 cc. volumetric flask and make up to the mark, adding a few drops of ether to destroy the bubbles. Mix thoroughly and pass through a dry filter. Titrate 250 cc. of the filtrate as directed under 3 (C) and calculate the per cent of arsenious oxid.

TOTAL ARSENIC OXID.⁷—OFFICIAL.

18

REAGENTS.

The reagents and solutions used are described under 3.

19

DETERMINATION.

Boil, on a hot plate or over a low flame, 2 grams of the sample with 5 cc. of concentrated nitric acid and 20 cc. of concentrated sulphuric acid in a Kjeldahl digestion flask or a covered casserole. After 10–15 minutes add fuming nitric acid or powdered sodium nitrate, in small quantities at a time, until all organic matter is destroyed and the solution is practically colorless. Cool, add about 50 cc. of water (to decompose any nitro-sulphuric acid formed) and heat again until all nitric acid fumes are expelled. Cool, transfer to a 250 cc. volumetric flask, make up to the mark with water, mix thoroughly, and filter through a dry filter.

Transfer 50 cc. of this filtrate to a 400 cc. Erlenmeyer flask, dilute with water to 100 cc., add 1 gram of potassium iodid,⁸ heat to boiling and evaporate to about 40 cc. (not less). Cool, dilute to 150–200 cc., and remove the excess of iodine with N/20 sodium thiosulphate. In case the solution is slightly colored from organic matter or from any cause other than free iodine, add the thiosulphate until it is nearly colorless, then a few drops of the starch indicator, and continue adding the thiosulphate slowly until the blue color just disappears. Continue at once as directed under 3 (C) beginning with "neutralize with sodium bicarbonate." Subtract from this reading the number of cc. of the standard iodine solution corresponding to the arsenious oxid obtained in 17. Calculate the per cent of arsenic oxid in the sample.

20

WATER-SOLUBLE ARSENIOS OXID.—TENTATIVE.

Proceed as directed under 12, slightly acidifying the aliquot employed with hydrochloric acid before adding the excess of sodium bicarbonate.

WATER-SOLUBLE ARSENIC OXID.—TENTATIVE.

21

REAGENTS.

The solutions and reagents used are described under 3.

22

DETERMINATION.

Transfer an aliquot, 250 cc., of the water extract, from 20, to a casserole, add 5 cc. of concentrated sulphuric acid, evaporate to a small volume and heat on a hot plate till white fumes of sulphuric acid appear. Cover the casserole and add 1-2 cc. of fuming nitric acid and again heat till the appearance of white fumes. Cool, add a little water and, in order to expel the last traces of nitric acid, once more evaporate till white fumes appear. Cool, dilute to about 100 cc. with water, add 1 gram of potassium iodid^e and sufficient sulphuric acid to make the total amount present about 5 cc. Boil until the volume is reduced to about 40 cc. Cool, dilute to about 200 cc., remove the excess iodine with N/20 sodium thiosulphate and proceed as directed under 3 (C) beginning with "neutralize with sodium bicarbonate." Correct for the amount of the standard iodine solution necessary to produce the same color, using the same reagents and volume. Subtract from the corrected titration reading the number of cc. of the standard iodine solution corresponding to the arsenious oxid, obtained in 20. Calculate the per cent of arsenic oxid present.

LEAD ARSENATE.

23

MOISTURE.—TENTATIVE.

(a) *Powder*.—Dry 2 grams to constant weight at 105°–110° C. and report the loss in weight as moisture.

(b) *Paste*.—Proceed as under (a), using 50 grams.

Grind the dry sample to a fine powder, mix well, transfer a small portion to a sample bottle and again dry for 1-2 hours at 105°–110°C., and use this anhydrous material for the determination of total lead oxid and total arsenic.

TOTAL LEAD OXID.

24

Method I.—Official.

Heat, on a hot plate, 0.6906 gram of the dry powdered sample with about 25 cc. of dilute nitric acid (1 to 4) in a 600 cc. beaker. If necessary, remove any insoluble residue by filtration. Dilute to at least 400 cc., heat nearly to boiling, add ammonium hydroxid to incipient precipitation, then dilute nitric acid (1 to 10) to redissolve the precipitate, adding 1-2 cc. in excess. Pipette into this solution, kept almost boiling, 50 cc. of a hot 10% potassium chromate solution, stirring constantly. Decant while hot through a weighed Gooch, previously heated at 140°–150°C., wash several times by decantation and then on the filter with boiling water until the washings are colorless. Dry the lead chromate at 140°–150°C. to constant weight. The weight of lead chromate multiplied by 100 gives the per cent of lead monoxid (PbO) in the dried sample.

The lead chromate precipitate may contain a small amount of lead arsenate which causes slightly high results. This error rarely amounts to more than 0.1–0.2%.

Method II.¹⁰—Tentative.

(Not applicable in the presence of calcium.)

25

REAGENT.

Acidified alcohol.—Mix water 100 parts; 95% alcohol 200 parts; and concentrated sulphuric acid 3 parts by volume.

26

DETERMINATION.

Heat, on a hot plate, 0.7360 gram of the dry powdered sample with about 25 cc. of dilute nitric acid (1 to 4) in a porcelain evaporating dish or casserole. Remove any insoluble residue by filtration. Add 3 cc. of concentrated sulphuric acid and evaporate on the hot plate to the appearance of white fumes. It is important that all nitric acid be expelled. Cool, add 50 cc. of water and about 100 cc. of 95% alcohol, let stand several hours (preferably over-night) and filter through a weighed Gooch crucible, previously washed with water, the acidified alcohol and 95% alcohol, and dried at 200°C. Wash the precipitate of lead sulphate in the crucible about 10 times with the acidified alcohol and then with 95% alcohol until free from sulphuric acid. Dry at 200°C. to constant weight, keeping the crucible covered to prevent loss by spattering. The weight of the lead sulphate multiplied by 100 gives the per cent of lead monoxid (PbO) in the dried sample.

TOTAL ARSENIC.

27

Method I.¹—Official.

Proceed as directed under 5, using an amount of the sample equal to the arsenic oxid equivalent of 500 cc. of the standard iodine solution and titrating a 200 cc. aliquot of the distillate. The number of cc. used of the standard iodine solution represents directly the total per cent of arsenic in the sample expressed as arsenic oxid (As_2O_3).

Method II.¹¹—Official.

(Not applicable in the presence of antimony.)

28

REAGENTS.

The reagents and solutions used are described under 3.

29

DETERMINATION.

Dissolve an amount of the powdered sample equal to the arsenic oxid equivalent of 400 cc. of the standard iodine solution, in dilute nitric acid in a porcelain casserole or evaporating dish. Add 5 cc. of concentrated sulphuric acid and heat on the hot plate to copious evolution of white fumes. Wash into a 200 cc. graduated flask with water, cool, make up to the mark and filter through a dry filter. Transfer 100 cc. of the filtrate to an Erlenmeyer flask and proceed as directed under 22, beginning with "add 1 gram of potassium iodide," to "Subtract from the corrected titration reading." The number of cc. of the standard iodine solution used, divided by 2, represents directly the per cent of total arsenic in the sample expressed as arsenic oxid (As_2O_3).

WATER-SOLUBLE ARSENIC OXID.—TENTATIVE.

30

REAGENTS.

The reagents and solutions used are described under 3.

31

DETERMINATION.

Treat 2 grams of the original sample, if in the form of a powder, or 4 grams, if a paste, as directed under 12 through "Filter through a dry filter."

Place 250–500 cc. of the *clear* filtrate in an Erlenmeyer flask, add 3 cc. of concentrated sulphuric acid and evaporate on a hot plate. When the volume is reduced to about 100 cc., proceed as directed under 22 to "Subtract from the corrected titration reading." Calculate and report as per cent of water-soluble arsenic oxid (As_2O_3).

CALCIUM ARSENATE.

32

TOTAL ARSENIC.—OFFICIAL.

Proceed as directed under 5, using an amount of the powdered sample equal to the arsenic oxid equivalent of 250 cc. of the standard iodine solution.

The number of cc. of the standard iodine solution used represents directly the total per cent of arsenic in the sample expressed as arsenic oxid (As_2O_3).

ZINC ARSENITE.

33

TOTAL ARSENIC.—OFFICIAL.

Proceed as directed under 5, using an amount of the powdered sample equal to the arsenious oxid equivalent of 500 cc. of the standard iodine solution and titrating a 200 cc. aliquot of the distillate. The number of cc. of the standard iodine solution used represents directly the per cent of total arsenic in the sample expressed as arsenious oxid (As_2O_3).

34

TOTAL ARSENIOUS OXID.—TENTATIVE.

Proceed as directed under 7 or 8.

COPPER CARBONATE.

35

COPPER OXID.—OFFICIAL.

Dissolve a weighed quantity of the substance in dilute nitric acid and determine copper as directed under 13 or 14.

BORDEAUX MIXTURE.

36

MOISTURE.—OFFICIAL.

(a) *Powder*.—Dry 2 grams to constant weight at 105°–110°C. and express the loss in weight as moisture.

(b) *Paste*.—Heat about 100 grams in an oven at 90–100°C. until dry enough to powder readily, and note the loss in weight. Powder this partially dried sample, and determine the remaining moisture in 2 grams as under (a). Determine carbon dioxide, as directed under 38, both in the original paste and in this partially dried sample. Calculate the total moisture by the following formula:

$M = a + (100 - a)(b + c) - d$ in which

M = per cent total moisture in original paste;

a = per cent loss in weight of original paste during first drying;

b = per cent loss in weight of partially dried paste during second drying;

c = per cent carbon dioxide remaining in partially dried paste after first drying;

d = per cent total carbon dioxide in original paste.

CARBON DIOXID.⁴²—OFFICIAL.

37

APPARATUS.

This consists of a 200 cc. Erlenmeyer flask closed with a 2-holed stopper; one of these holes is fitted with a dropping funnel the stem of which extends almost to the bottom of the flask; the outlet of a condenser, which is inclined upward at an angle of 30° from the horizontal, passes downward through the other hole. The upper end of the condenser is connected with a calcium chlorid tube which in turn is connected with a double U-tube filled in the middle with pumice fragments, previously saturated with copper sulphate solution and subsequently dehydrated, and with calcium chlorid at either end. Then follow 2 weighed U-tubes for absorbing the carbon dioxid, the first filled with porous soda-lime, and the second, one third with soda-lime and two thirds with calcium chlorid, the latter reagent being placed at the exit end of the train. A Geissler bulb, partly filled with sulphuric acid, is attached to the last U-tube to show the rate of gas flow. An aspirator is connected with the Geissler bulb to draw air through the apparatus. An absorption tower filled with soda-lime is connected with the mouth of the dropping funnel to remove carbon dioxid from the air entering the apparatus.

38

DETERMINATION.

Weigh 2 grams of the powder or 10 grams of the paste into the Erlenmeyer flask, add about 20 cc. of water, attach the flask to the apparatus omitting the 2 weighed U-tubes, and draw carbon dioxid-free air through the apparatus until the original air is displaced. Then attach the weighed U-tubes in the position as described in 37, close the stop-cock of the dropping funnel, fill half full with dilute hydrochloric acid (1 to 1), reconnect with the soda-lime tower, and allow the acid to flow into the Erlenmeyer flask, slowly if there is much carbon dioxid, rapidly if there is little. When effervescence diminishes, place a low Bunsen flame under the flask and start a flow of water through the condenser, a slow current of air being allowed to flow through the apparatus at the same time. Maintain a steady but quiet ebullition, and a slow air current through the apparatus. Boil for a few minutes after the water has begun to condense in the condenser, then remove the flame and continue the aspiration of air at the rate of about 2 bubbles per second until the apparatus is cool. Disconnect the tared absorption tubes, cool in the balance case and weigh. The increase in weight is due to carbon dioxid.

COPPER.

39

Electrolytic Method.—Official.

Dissolve 2 grams of the dry powdered sample in 20 cc. of water and 5 cc. of concentrated nitric acid, dilute to 100 cc., wash into a weighed 150 cc. platinum dish, and electrolyze, using a rotating spiral anode and a current of about 3 amperes. After all the copper is deposited (requiring about 30 minutes), wash the deposit with water by siphoning, then rinse with alcohol, dry for a few minutes in an oven, and weigh. Calculate the per cent of copper in the sample.

40

Thiosulphate Method.—Official.

Dissolve 2 grams of the dry powdered sample in about 50 cc. of 10% nitric acid, add ammonium hydroxid solution in excess and heat; then, without removing the precipitate which is formed, boil off the excess of ammonia, add 5-10 cc. of acetic acid, cool, add 10 cc. of 30% potassium iodid solution, and titrate as directed under VIII, 29.

BORDEAUX MIXTURE WITH PARIS GREEN.**41** **MOISTURE.—OFFICIAL.**

Proceed as directed under **36**.

42 **CARBON DIOXID.—OFFICIAL.**

Proceed as directed under **38**.

COPPER.**43** *Method I.—Tentative.*

Dissolve 2 grams of the dry powdered sample in a few cc. of strong nitric acid, add 25 cc. of a 3% solution of hydrogen peroxid and warm for 5–10 minutes. Make slightly alkaline with ammonium hydroxid and then slightly acid again with dilute nitric acid. Transfer to a weighed 150 cc. platinum dish, add 15–20 cc. of hydrogen peroxid, dilute to 100 cc. and electrolyze, using a rotating spiral anode and a current not exceeding 2 amperes. After the electrolysis has proceeded for about 20 minutes, add to the electrolyte 0.5 gram of ferric sulphate dissolved in a few cc. of water together with a drop or two of nitric acid. After all the copper is deposited, wash the deposit with water by siphoning, then rinse with alcohol, dry for a few minutes in an oven, weigh and calculate the per cent of copper. (Do not pass the current for more than 5–10 minutes after all the copper has been deposited without adding more ferric sulphate solution.)

44 *Method II.—Tentative.*

Treat 1 gram of the dry powdered sample with 20 cc. of water and 5–6 cc. of concentrated nitric acid, heat to boiling, cool, and add a slight excess of concentrated ammonium hydroxid. Wash the solution and precipitate into a weighed platinum dish of about 150 cc. capacity, and electrolyze, using a rotating anode and a current of about 4 amperes and 3–4 volts for about 90 minutes (or until all the copper is deposited). Wash the deposit by siphoning until the deposit is clean, being careful not to use too much wash water. Dissolve the copper in 5 cc. of concentrated nitric acid, dilute to 100 cc. and electrolyze as before, except that all the copper will be deposited in 30 minutes. Wash the deposit with water by siphoning, then rinse with alcohol, dry for a minute or so in an oven, weigh and calculate the per cent of copper.

45 **TOTAL ARSENIC.—OFFICIAL.**

Proceed as directed under **5**, using an amount of the dry powdered sample equal to the arsenious oxid equivalent of 500 cc. of the standard iodine solution. The number of cc. of the standard iodine solution used, divided by 2, represents directly the per cent of total arsenic in the sample expressed as arsenious oxid (As_2O_3).

TOTAL ARSENIOUS OXID.**46** *Method I.—Tentative.*

Proceed as directed under **7**, using an amount of the dry, powdered sample equal to the arsenious oxid equivalent of 200 cc. of the standard iodine solution. Before titrating, all the copper must be in solution. The corrected number of cc. of the standard iodine solution used, divided by 2, represents directly the per cent of total arsenious oxid (As_2O_3) in the sample.

47 *Method II.—Tentative.*

Proceed as directed under **8**.

48 **WATER-SOLUBLE ARSENIUS OXID.—TENTATIVE.**

Proceed as directed under **20**, using 2 grams of the sample.

BORDEAUX MIXTURE WITH LEAD ARSENATE.**49** **MOISTURE.—OFFICIAL.**

Proceed as directed under **36**.

50 **CARBON DIOXID.—OFFICIAL.**

Proceed as directed under **38**.

51 **COPPER.—TENTATIVE.**

Proceed as directed under **44**.

52 **LEAD OXID.—TENTATIVE.**

Dissolve the lead peroxid (which will contain a little arsenic) from the anodes used in the copper electrolysis, under **51**, by means of dilute nitric acid and a little hydrogen peroxid, and add to this solution the washings from both electrolyses of copper. Add ammonium chlorid to dissolve any lead sulphate which may have precipitated out and make the solution up to 1 liter. Concentrate a 500 cc. aliquot of this solution to about 300 cc. (all hydrogen peroxid must be expelled from the solution), transfer to a 400 cc. beaker and precipitate the lead as lead chromate as directed under **24**.

53 **TOTAL ARSENIC.—OFFICIAL.**

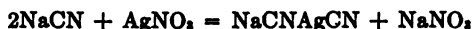
Proceed as directed under **5**, using an amount of the dry, powdered sample equal to the arsenic oxid equivalent of 500 cc. of the standard iodine solution. The number of cc. of the standard iodine solution used, divided by 2, represents directly the per cent of total arsenic in the sample expressed as arsenic oxid (As_2O_3).

54 **WATER-SOLUBLE ARSENIC OXID.—TENTATIVE.**

Proceed as directed under **31**.

SODIUM AND POTASSIUM CYANIDS.**55** **CYANOGEN.—OFFICIAL.**

Weigh about 10 grams of the sample in a weighing bottle, dissolve in water, and make up to volume in a liter graduated flask. To a 50 cc. aliquot add N/20 silver nitrate, drop by drop, stirring constantly, until 1 drop produces a permanent turbidity. In calculating the results, 1 equivalent of silver is equal to 2 equivalents of cyanogen, according to the following equation:



Reserve the titrated solution for the determination of chlorin under **56**.

56

CHLORIN.¹⁴—OFFICIAL.

After completion of the titration for cyanogen, as directed under 55, add a few cc. of 10% potassium chromate solution as indicator and titrate with N/20 silver nitrate until the appearance of the red-brown color of silver chromate.

The first titration with silver nitrate represents the cyanogen present according to the equation above. The second titration represents the cyanogen and chlorin according to the following equation: $\text{NaCNAgCN} + \text{NaCl} + 2\text{AgNO}_3 = 2\text{NaNO}_3 + 2\text{AgCN} + \text{AgCl}$. Therefore the second minus the first reading represents the chlorin present in terms of silver nitrate.

SOAP.

MOISTURE.

57

*Modified Method of Benedickt and Lewkowitsch.*¹⁵—Tentative.

Weigh about 5 grams of the sample in a tared, 100 cc. beaker, in which is previously placed a $\frac{1}{2}$ inch layer of recently ignited, dry sand, and a small glass rod; if the soap is hard, cut off the soap in very thin strips. Add 25 cc. of alcohol, or more if necessary, and dissolve on the water bath, stirring constantly. Evaporate the alcohol, heat in an oven at 110°C. until the soap is nearly dry, and weigh, then dry again for 30 minutes and weigh. Continue this alternate drying and weighing until the weight changes only a few milligrams during the course of 30 minutes' drying.

58

POTASSIUM AND SODIUM.¹⁶—TENTATIVE.

Dissolve about 5 grams of the soap in water; decompose with hydrochloric acid, filter off the water and wash the fat with cold water. Determine both potassium and sodium in the filtrate as directed under II, 21.

SODA LYE.

59

CARBONATE AND HYDROXID.¹⁷—OFFICIAL.

Weigh about 10 grams of the sample from the weighing bottle, dissolve in carbon dioxid-free water and make up to a definite volume. Titrate an aliquot of this solution with N/2 hydrochloric acid, using methyl orange as an indicator, and note the total alkalinity thus found. Transfer an equal aliquot to a graduated flask and add enough barium chlorid solution to precipitate all the carbonate, avoiding any unnecessary excess. Dilute to the mark with carbon dioxid-free water, stopper, shake, and set aside. When the liquid becomes clear, pipette off one half and titrate with N/2 hydrochloric acid, using phenolphthalein as an indicator. The number of cc. of N/2 acid, required for this titration, multiplied by 2 gives the number of cc. of N/2 acid required to neutralize the sodium hydroxid present in the original aliquot. The difference between this figure and the number of cc. of N/2 hydrochloric acid required for the total alkalinity represents the number of cc. of N/2 acid required to neutralize the sodium carbonate present in the aliquot. Calculate the percentages of sodium carbonate and hydroxid present in the sample.

TOBACCO AND TOBACCO EXTRACT.

NICOTIN.

Kissling Method.—Official.

60

REAGENTS.

(a) *Alcoholic sodium hydroxid solution.*—Dissolve 6 grams of sodium hydroxid in 40 cc. of water and 60 cc. of 90% alcohol.

- (b) 0.4% sodium hydroxid solution.
- (c) N/10 sulphuric acid.—One cc. is equivalent to 16.22 mg. of nicotin.
- (d) Phenacetolin solution.—Prepare a 0.5% alcoholic solution.
- (e) Cochineal solution.—Prepare as directed under I, 16 (k).

61

DETERMINATION.

Weigh 5–6 grams of tobacco extract, or 20 grams of finely powdered tobacco which has been previously dried at 60°C. if necessary, into a small beaker. Add 10 cc. of the alcoholic sodium hydroxid and follow, in the case of tobacco extract, with enough pure powdered calcium carbonate to form a moist but not lumpy mass. Mix thoroughly, transfer to a Soxhlet extractor and exhaust for about 5 hours with ether. Evaporate the ether at a low temperature, and take up the residue with 50 cc. of the 0.4% sodium hydroxid solution. Transfer this residue by means of water to a 500 cc. Kjeldahl flask, and distil with steam, passing the distillate through a condenser cooled by a rapidly flowing current of water. Use a 3-bend outflow tube, and, to prevent bumping and frothing, add a few pieces of pumice, and a small piece of paraffin. Distil till all the nicotin has passed over, the distillate usually varying from 400–500 cc. When completed, only about 15 cc. of the liquid should remain in the flask. Titrate the distillate with N/10 sulphuric acid, using the phenacetolin or cochineal solution as indicator.

Silicotungstic Acid Method.^{1a}—Official.

62

REAGENTS.

- (a) *Silicotungstic acid solution*.—Prepare a 12% solution of the silicotungstic acid having the following formula: $4\text{H}_2\text{O} \cdot \text{SiO}_2 \cdot 12\text{WO}_3 \cdot 22\text{H}_2\text{O}$.
- (b) *Sodium or potassium hydroxid solution (1 to 2)*.
- (c) *Dilute hydrochloric acid (1 to 4)*.

63

DETERMINATION.

Weigh such an amount of the preparation as will contain preferably between 0.1 and 1.0 gram of nicotin (if the sample contains very little nicotin, about 0.1%, do not increase the amount to the point where it interferes with the distillation); wash with water into a 500 cc. round-bottomed distillation flask; add a little paraffin to prevent frothing, a few small pieces of pumice and a slight excess of the sodium or potassium hydroxid, using phenolphthalein as an indicator. Distil rapidly in a current of steam through a well-cooled condenser, connected by means of an adapter with a suitable flask containing 10 cc. of the dilute hydrochloric acid. When distillation is well under way, heat the distillation flask to reduce the volume of the liquid as far as practicable without bumping or undue separation of insoluble matter. Distil until a few cc. of the distillate show no cloud or opalescence when treated with a drop of the silicotungstic acid and a drop of the dilute hydrochloric acid. Confirm the alkalinity of the residue in the distillation flask with phenolphthalein solution. Make up the distillate, which may amount to 1000–1500 cc., to a convenient volume (the solution may be concentrated on the steam bath without loss of nicotin), mix well and pass through a large dry filter if not clear. Test a portion with methyl orange to assure its acidity. Pipette an aliquot, containing about 0.1 gram of nicotin, into a beaker (if the samples contain very small amounts of nicotin, an aliquot containing as little as 0.01 gram of nicotin may be used), add to each 100 cc. of liquid 3 cc. of the dilute hydrochloric acid, or more if the necessity is indicated by the test with methyl orange, and add 1 cc. of the silicotungstic acid for each 0.01 gram of nico-

tin supposed to be present. Stir thoroughly and let stand overnight. Before filtering, stir the precipitate to see that it settles quickly and is in crystalline form; then filter on an ashless filter paper, and wash with cold dilute hydrochloric acid (1 to 1000). Transfer the paper and precipitate to a weighed platinum crucible, dry carefully, and ignite until all carbon is destroyed. Finally heat over a Teclu or Meker burner for not more than 10 minutes. The weight of the residue multiplied by 0.114 gives the weight of nicotin present in the aliquot.

FORMALDEHYDE SOLUTIONS.

FORMALDEHYDE.

*Hydrogen Peroxid Method.*¹⁴—Official.

64

REAGENTS.

- (a) *N/1 sulphuric acid.*
- (b) *N/1 sodium hydroxid.*—One cc. is equivalent to 30.02 mg. of formaldehyde.
- (c) *Hydrogen peroxid.*—An approximately 3% solution. If the hydrogen peroxid solution is acid, neutralize with (b), using litmus solution as indicator.
- (d) *Litmus solution.*—A solution of purified litmus.

65

DETERMINATION.

Measure 50 cc. of *N/1* sodium hydroxid into a 500 cc. Erlenmeyer flask and add 50 cc. of the hydrogen peroxid. Then add 3 grams of the formaldehyde solution under examination, allowing the point of the pipette to reach nearly to the liquid in the flask. Place a funnel in the neck of the flask and heat on the steam bath for 5 minutes, shaking occasionally. Remove from the steam bath, wash the funnel with water, cool the flask to about room temperature, and titrate with *N/1* acid, using the litmus solution as indicator. It is necessary to cool the flask before titration with the acid to get a sharp end point with the litmus. Calculate the per cent of formaldehyde.

*Cyanid Method.*²⁰—Official.

66

REAGENTS.

- (a) *N/10 silver nitrate.*
- (b) *N/10 ammonium sulphocyanate.*
- (c) *Potassium cyanid solution.*—Dissolve 3.1 grams of potassium cyanid in 500 cc. of water.
- (d) *50% nitric acid.*

67

DETERMINATION.

Treat 15 cc. of the *N/10* silver nitrate with 6 drops of the 50% nitric acid in a 50 cc. volumetric flask; add 10 cc. of the potassium cyanid solution, dilute to the mark, shake well, filter through a dry filter and titrate 25 cc. of the filtrate with *N/10* ammonium sulphocyanate as directed under **III, 15**. Acidify another 15 cc. portion of the *N/10* silver nitrate with 6 drops of the 50% nitric acid and treat with 10 cc. of the potassium cyanid solution to which has been added a measured quantity (the weight of which must be calculated from the specific gravity) of the formaldehyde solution containing not over 2.5 grams of a 1% solution or the equivalent. Make up to 50 cc., filter and titrate a 25 cc. aliquot with the *N/10* ammonium sulphocyanate for the excess of silver as before. The difference between the number of cc. of *N/10* ammonium sulphocyanate used in these 2 titrations, multiplied

by 2, gives the number of cc. of N/10 ammonium sulphocyanate corresponding to the potassium cyanid used by the formaldehyde. Calculate the per cent of formaldehyde present (1 cc. of N/10 ammonium sulphocyanate is equivalent to 3 mg. of formaldehyde (HCHO)).

LIME-SULPHUR SOLUTIONS.²¹

TOTAL SULPHUR.—OFFICIAL.

68

PREPARATION OF SOLUTION.

Weigh 10 grams of the solution and dilute to the mark in a 250 cc. graduated flask with recently boiled and cooled water.

69

DETERMINATION.

Transfer a 10 cc. aliquot to a 400 cc. beaker, add about 3 grams of sodium peroxid, cover immediately with a watch glass and warm on the steam bath, with frequent shaking, until all the sulphur is oxidized to sulphate, adding more sodium peroxid if necessary. Dilute, acidify with hydrochloric acid, evaporate to dryness, treat with water acidified with hydrochloric acid, boil, and filter to remove silica, if present. Dilute the filtrate to 300 cc., add 50 cc. of concentrated hydrochloric acid,²² heat to boiling, and precipitate with 10% barium chlorid solution slowly and stirring constantly. (The rate is best regulated by attaching a suitable capillary tip to the burette containing the barium chlorid solution.) Evaporate to dryness on the steam bath, take up with hot water, filter through a quantitative filter paper, wash until free from chlorin, ignite and heat to constant weight over a Bunsen burner. Calculate the sulphur from the weight of barium sulphate. Previous to use test the reagents for sulphur and, if present, make corrections accordingly.

SULPHID SULPHUR.—OFFICIAL.

70

REAGENT.

Ammoniacal zinc solution.—Dissolve 50 grams of pure zinc chlorid in water, add ammonium hydroxid in sufficient quantity to redissolve the precipitate first formed, then add 50 grams of ammonium chlorid²³ and dilute to 1 liter.

71

DETERMINATION.

Dilute 10 cc. of the solution, prepared as directed under 68, to about 100 cc. and add the ammoniacal zinc solution until the sulphid is all precipitated, indicated by the addition of a drop of the clear solution to a few drops of nickel sulphate solution. Filter immediately, wash the precipitate thoroughly with cold water and transfer it and the filter paper to a beaker. Cover with water, disintegrate with a glass rod and add about 3 grams of sodium peroxid, keeping the beaker well covered with a watch glass. Warm on the steam bath with frequent shaking until all the sulphur is oxidized to sulphate, adding more sodium peroxid if necessary. Make slightly acid with hydrochloric acid, filter to remove shreds of filter paper, wash thoroughly with hot water, and determine the sulphur in the filtrate exactly as under 69.

72

THIOSULPHATE SULPHUR.—OFFICIAL.

Dilute 50 cc. of the solution, prepared as under 68, to about 100 cc. in a 200 cc. graduated flask. Add a slight excess of the ammoniacal zinc chlorid and dilute to the mark. Shake thoroughly and filter through a dry filter. To 100 cc. of the filtrate add a few drops of methyl orange and exactly neutralize with N/10 hydrochloric

acid. Titrate this neutral solution with approximately N/20 iodine, 3 (c), using a few drops of starch solution as indicator. From the number of cc. of iodine solution used, calculate the thiosulphate sulphur present.

73

SULPHATE SULPHUR.—OFFICIAL.

To the solution from the determination in 72, add 2 or 3 drops of hydrochloric acid, precipitate in the cold with 10% barium chloride solution, allow to stand overnight, filter, calculate the sulphur from the weight of barium sulphate and report as sulphate sulphur.

74

TOTAL LIME.—OFFICIAL.

To 25 cc. of the solution, prepared as under 68, add 10 cc. of concentrated hydrochloric acid, evaporate to dryness on the steam bath, treat with water and a little hydrochloric acid, warm until all the calcium chloride is dissolved, and filter from sulphur and any silica that may be present. Oxidize the filtrate by boiling with a little concentrated nitric acid, make ammoniacal, filter from iron and aluminium if present, heat to boiling and precipitate the calcium with ammonium oxalate solution. Filter, wash and ignite over a blast lamp to constant weight; weigh the residue as calcium oxide.

BIBLIOGRAPHY.

- ¹ J. Ind. Eng. Chem., 1916, 8: 327.
- ² Ibid., 1909, 1: 208.
- ³ J. Assoc. Official Agr. Chemists, 1915, 1: 436, 446.
- ⁴ J. Am. Chem. Soc., 1901, 23: 115.
- ⁵ Ibid., 1902, 24: 1082.
- ⁶ Ibid., 1900, 22: 802.
- ⁷ U. S. Bur. Chem. Bull. 122, p. 106.
- ⁸ Am. J. Sci., 1890, 3rd ser., 40: 68.
- ⁹ U. S. Bur. Chem. Bull. 137, p. 40.
- ¹⁰ Ibid., 105, p. 166.
- ¹¹ Ibid., p. 167.
- ¹² Fresenius. Quantitative Chemical Analysis. Revised and amplified translation of the 6th German ed., 2: 1180; U. S. Geol. Surv. Bull. 422, p. 179.
- ¹³ Sutton. Volumetric Analysis. 10th ed., 1911, p. 207.
- ¹⁴ Ibid., 9th ed., rev., p. 201.
- ¹⁵ Lewkowitsch. Chemical Technology and Analysis of Oils, Fats and Waxes. 5th ed., 1915, 3: 348.
- ¹⁶ Ibid., 346.
- ¹⁷ Sutton. Volumetric Analysis. 10th ed., 1911, p. 61.
- ¹⁸ U. S. Bur. Animal Industry, Bull. 133.
- ¹⁹ Ber., 1898, 31: 2979; J. Am. Chem. Soc., 1905, 27: 1183; U. S. Bur. Chem. Bull. 99, p. 30; 132, p. 49; 137, p. 47.
- ²⁰ Z. anal. Chem., 1897, 36: 18; U. S. Bur. Chem. Bull. 132, p. 49.
- ²¹ J. Assoc. Official Agr. Chemists, 1915, 1: 76.
- ²² J. Am. Chem. Soc., 1911, 33: 844.
- ²³ J. Soc. Chem. Ind., 1912, 31: 369.

VIII. FOODS AND FEEDING STUFFS.

1

PREPARATION OF SAMPLE.—OFFICIAL.

Grind the sample so that it will pass through a sieve having circular openings $\frac{1}{16}$ inch (1 mm.) in diameter. If the sample can not be ground, reduce it to as fine a state as possible.

MOISTURE.

2

Direct Drying.—Official.

Dry a quantity of the substance, representing about 2 grams of dry material, in a current of dry hydrogen or in vacuo at the temperature of boiling water to constant weight (approximately 5 hours). If the substance be held in a glass vessel, the latter should not be in contact with the boiling water.

3

Drying in Vacuo without Heat.—Tentative.

Mix the sample thoroughly and weigh by difference 2-5 gram portions from a stoppered weighing bottle into tared, covered crucibles. Where subsequent fat determinations are to be made, fat extraction cones may be used. Substances that dry down to horn-like material should be mixed with fat-free cotton or other suitable material (previously tared with the container). Place 200 cc. of fresh concentrated sulphuric acid in a strong, tight 6 inch vacuum desiccator. Put triplicate samples in separate desiccators, and exhaust by means of a vacuum pump. If a pump is not available, place 10 cc. of ether in a small beaker in the desiccator, and exhaust with a water filter pump.

Between the pump and the desiccator interpose an empty bottle, next to the desiccator, and a bottle of water. Draw the air from the desiccator through the water and turn the desiccator stop-cock at just the instant when the water begins to rise in the tube leading from the empty bottle.

Gently rotate the desiccator 4 or 5 times during the first 12 hours to mix the sulphuric acid with the water which has collected as an upper layer. At the end of 24 hours open the desiccator, forcing the incoming air to bubble through concentrated sulphuric acid, and make the first weighing. After weighing place in a desiccator containing fresh concentrated sulphuric acid and exhaust as before. Rotate the desiccator several times during the interval and weigh again after a suitable period of drying. Repeat this process of drying in vacuo over sulphuric acid until the weight is constant.

4

ASH.—OFFICIAL.

Char a quantity of the substance, representing about 2 grams of the dry material, and burn until free from carbon at a low heat, not to exceed dull redness. If a carbon-free ash can not be obtained in this manner, exhaust the charred mass with hot water, collect the insoluble residue on a filter, burn till the ash is white or nearly so, and then add the filtrate to the ash and evaporate to dryness. Heat to low redness till the ash is white or grayish white and weigh.

5

CRUDE PROTEIN.—OFFICIAL.

Determine nitrogen as directed under I, 18, 21, or 23, and multiply the result by 6.25.

ALBUMINOID NITROGEN.—OFFICIAL.

6

REAGENT.

Stutzer's reagent.—Prepare cupric hydroxid as follows: Dissolve 100 grams of pure copper sulphate in 5 liters of water, add 2.5 cc. of glycerol, and then dilute sodium hydroxid solution until the liquid is just alkaline; filter, rub the precipitate up with water containing 5 cc. of glycerol per liter, and wash by decantation or filtration until the washings are no longer alkaline. Rub the precipitate up again in a mortar with water containing 10% of glycerol, thus preparing a uniform gelatinous mass that can be measured with a pipette. Determine the quantity of copper hydroxid per cc. of this mixture.

7

DETERMINATION.

Place 0.7 gram of the substance in a beaker, add 100 cc. of water, and heat to boiling; or, in case of substances rich in starch, heat on the water bath for 10 minutes; add a quantity of the Stutzer's reagent containing about 0.5 gram of the hydroxid; stir thoroughly, filter when cold, wash with cold water, and, without removing the precipitate from the filter, determine the nitrogen according to I, 18, 21 or 23, adding sufficient potassium sulphid solution to completely precipitate all of the copper and mercury. The filter paper used must be practically free from nitrogen. If the material (such as seeds, seed residue, or oil cake) is rich in alkaline phosphates, add, to decompose the alkaline phosphates, 1–2 cc. of a concentrated potash or soda alum solution, free from ammonia, then the copper hydroxid, and mix well by stirring. If this is not done, copper phosphate and free alkali may be formed, and the protein-copper precipitate partially dissolved in the alkaline liquid.

8

AMIDO NITROGEN.—OFFICIAL.

Subtract the amount of albuminoid nitrogen from the amount of total nitrogen to obtain the amido nitrogen.

CRUDE FAT OR ETHER EXTRACT.

Direct Method.—Official.

9

REAGENT.

Anhydrous ether.—Wash any of the commercial brands of ether with 2 or 3 successive portions of water, add solid sodium or potassium hydroxid, and let stand until most of the water has been abstracted from the ether. Decant into a dry bottle, add small pieces of carefully cleaned metallic sodium, and let stand until there is no further evolution of hydrogen gas. Keep the ether, thus dehydrated, over metallic sodium in lightly stoppered bottles.

10

DETERMINATION.

Large quantities of soluble carbohydrates may interfere with the complete extraction of the fat. In such cases extract with water before proceeding with the determination. Extract about 2 grams of material, dried as under 2 or 3, with the anhydrous ether for 16 hours. Dry the extract at the temperature of boiling water for 30 minutes, cool in a desiccator, and weigh; continue, at 30 minutes intervals, this alternate drying and weighing to constant weight. For most feeds a period of 1–1½ hours is required.

11

Indirect Method.—Official.

Determine the moisture, as directed in 2 or 3, then extract the dried substance for 16 hours as directed under 10, dry again and regard the loss of weight as ether extract.

SUCROSE.

OPTICAL METHODS.

12

GENERAL DIRECTIONS FOR RAW SUGARS.—TENTATIVE.

(Rules¹ of the International Commission for Unifying Methods of Sugar Analysis.)

"In general all polarizations are to be made at 20°C."

"The verification of the saccharimeter must also be made at 20°C. For instruments using the Ventske scale 26 grams of pure dry sucrose, weighed in air with brass weights, dissolved in 100 metric cc. at 20°C. and polarized in a room, the temperature of which is also 20°C., must give a saccharimeter reading of exactly 100.00. The temperature of the sugar solution during polarization must be kept constant at 20°C."

"For countries where the mean temperature is higher than 20°C., saccharimeters may be adjusted at 30°C. or any other suitable temperature, under the conditions specified above, provided that the sugar solution be made up to volume and polarized at this same temperature."

"In effecting the polarization of substances containing sugar employ only half-shade instruments." The saccharimeter used can be either single or double wedge and should be a half-shadow instrument with either double or triple field.

"During the observation keep the apparatus in a fixed position and so far removed from the source of light that the polarizing Nicol is not warmed."

"As sources of light employ lamps which give a strong illumination such as triple gas burner with metallic cylinder, lens and reflector; gas lamps with Auer (Welsbach) burner; electric lamp; petroleum duplex lamp; sodium light." Whenever there is any irregularity in the sources of light such as that due to the convolutions of the filament in the case of electric light or to the meshes of the gauze in the case of the Welsbach light, place a thin ground-glass plate between the source of light and the polariscope so as to render the illumination uniform.

"Before and after each set of observations the chemist must satisfy himself of the correct adjustment of his saccharimeter by means of standardized quartz plates. He must also previously satisfy himself of the accuracy of his weights, polarization flasks, observation tubes and cover-glasses. (Scratched cover-glasses must not be used.) Make several readings and take the mean thereof, but no one reading may be neglected." Such plates are standardized to read to the second decimal point and by their use a quick and at the same time accurate test can be made. In using such plates for testing saccharimeters, it is necessary that the instrument, as well as the plate, be at 20°C. before making a reading. Different points of the scale, preferably 20°, 50°, 80°, and 100°, (sugar scale) should be tested against the plates.

"In making a polarization use the whole normal weight for 100 cc. or a multiple thereof for any corresponding volume."

"As clarifying and decolorizing agents use either basic acetate of lead, alumina cream, or concentrated solution of alum. Boneblack and decolorizing powders are to be excluded." Whenever reducing sugars are determined in the solution for polarizing, use only neutral lead acetate for clarification as basic lead acetate causes precipitation of some of the reducing sugars. In addition to these clarifying agents,

neutral lead acetate and basic lead nitrate (Herles' solution) have been made official by the Association.

"After bringing the solution exactly to the mark at the proper temperature, and after wiping out the neck of the flask with filter paper, pour all of the well-shaken clarified sugar solution on a rapidly acting filter. Reject the first portions of the filtrate, and use the rest, which must be perfectly clear, for polarization." It is advisable to reject the first 20 cc. that run through, then cover the funnel with a watch glass and use the remainder for polarization. In no case should the whole solution or any part be returned to the filter. If cloudy after the 20 cc. have been rejected, begin a new determination.

"Whenever white light is used in polarimetric determinations, the same must be filtered through a solution of potassium dichromate of such a concentration that the percentage content of the solution multiplied by the length of the column of the solution in centimeters is equal to nine." This concentration must be doubled in reading carbohydrate materials of high rotation dispersion, such as commercial glucose, etc.

13

PREPARATION AND USE OF CLARIFYING REAGENTS.—TENTATIVE.

(a) *Basic lead acetate solution*.—Boil 430 grams of neutral lead acetate, 130 grams of litharge, and 1 liter of water for 30 minutes. Allow the mixture to cool and settle and dilute the supernatant liquid to a specific gravity of 1.25 with recently boiled water. Solid basic lead acetate may be substituted for the normal salt and litharge in the preparation of the solution.

(b) *Alumina cream*.—Prepare a cold saturated solution of alum in water. Add ammonium hydroxid with constant stirring until the solution is alkaline to litmus, allow the precipitate to settle and wash by decantation with water until the wash water gives only a slight test for sulphates with barium chlorid solution. Pour off the excess of water and store the residual cream in a stoppered bottle.

(c) *Dry basic lead acetate (Horne method)*.—This clarifying agent is obtained as a dry powdered salt and should contain 72.8% of lead, which corresponds to a composition of $3\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{PbO}$. Dissolve the normal or half-normal weight of the sugar solution in a flask with water and complete the volume. Add a small quantity of the dry salt and shake, then add more and shake again, repeating until completely precipitated but avoiding any excess. Of this salt 0.1346 gram is equivalent to 1 cc. of the basic lead acetate solution, described under (a). When molasses or any other substance producing a heavy precipitate is being clarified, some dry, coarse sand should be added to break up the balls of basic lead acetate and the precipitate. (This method is to have equal weight with the use of a solution of basic lead acetate in clarifying cane, sorghum, and beet products.)

(d) *Neutral lead acetate*.—Prepare a saturated solution of neutral lead acetate and add it to the sugar solution before completing to volume. Its use is imperative when determining the reducing sugars in the solution used for polarization.

(e) *Basic lead nitrate (Herles' solution)*.—(1) Dissolve 250 grams of lead nitrate in water and make up to 500 cc. (2) Dissolve 25 grams of sodium hydroxid in water and make up to 500 cc.

Add equal amounts of (1) and (2) to the sugar solution, shake, and add more if complete precipitation has not occurred, but avoid any excess. Then complete the volume with water. When this solution is used for clarification, the factor in the Clerget determination becomes 143.5 instead of 142.66.

DETERMINATION OF SUCROSE IN THE ABSENCE OF RAFFINOSE.

(In the presence of much levulose, as in honeys and fruits products, the optical method for sucrose gives too high a result.)

14 *By Polarization Before and After Inversion with Hydrochloric Acid.—Official.*

Dissolve the normal weight (26 grams) of the substance in water, add basic lead acetate carefully, avoiding any excess, then 1-2 cc. of alumina cream, shake, and dilute to 100 metric cc., filter, rejecting the first 20 cc. of the filtrate, cover the filter with a watch glass and, when sufficient filtrate is collected, polarize in a 200 mm. tube. The reading so obtained is the direct reading (P of formula given below) or polarization before inversion. For the invert reading, remove the lead from the solution either (1) by adding anhydrous potassium oxalate, a little at a time, to the remaining solution, avoiding an excess and removing the precipitated lead by filtration; or, (2) by adding anhydrous sodium carbonate under the same conditions. Introduce 50 cc. of the lead-free filtrate into a 100 cc. flask (if sodium carbonate was used for removing the lead, neutralize carefully the excess of sodium carbonate with a few drops of dilute hydrochloric acid) and add 25 cc. of water. Then add, little by little, while rotating the flask, 5 cc. of hydrochloric acid, (sp. gr. 1.20). Heat the flask after mixing, in a water bath kept at 70°C. The temperature of the solution in the flask should reach 67°-69°C. in 2½-3 minutes. Maintain a temperature of as nearly 69°C. as possible for 7-7½ minutes, making the total time of heating 10 minutes. Remove the flask and cool the contents rapidly to 20°C. and dilute to 100 cc. Polarize this solution in a tube provided with a lateral branch and a water jacket, maintaining a temperature of 20°C. This reading must be multiplied by 2 to obtain the invert reading. If it is necessary to work at a temperature other than 20°C., which is allowable within narrow limits, the volumes must be completed and both direct and invert polarizations must be made at exactly the same temperature.

The inversion may also be accomplished as follows: (1) To 50 cc. of the clarified solution, freed from lead, add 5 cc. of hydrochloric acid (sp. gr. 1.20) and set aside for 24 hours at a temperature not below 20°C.; or, (2) If the temperature be above 25°C. set aside for 10 hours. Make up to 100 cc. at 20°C. and polarize as directed above.

Calculate sucrose by one of the following formulas:

For substances in which the invert solution contains more than 1½ grams of invert sugar per 100 cc.—The following formula is to be used when substances like raw sugars are polarized:

$$S = \frac{100 (P - I)}{142.66 - \frac{T}{2}} \text{ in which}$$

S = per cent of sucrose;

P = direct reading normal solution;

I = invert reading normal solution;

T = temperature at which readings are made.

For substances in which the concentration of the invert solution is less than 1½ grams per 100 cc.—The following formula, which takes into account the concentration of the sugar in solution, should be used in all other cases.

$$S = \frac{100 (P - I)}{142.66 - \frac{T}{2} - 0.0065 \left[142.66 - \frac{T}{2} - (P - I) \right]} \text{ in which}$$

S = per cent of sucrose;
 P = direct reading normal solution;
 I = invert reading normal solution;
 T = temperature.

By Polarization Before and After Inversion with Invertase.—Tentative.

15

REAGENT.

Invertase solution (Hudson Method.³)—Mix 1 kilo of pressed baker's or brewer's yeast with 1 liter of tap water and 50 cc. of toluene and keep at room temperature 2–3 days to allow autolysis to proceed to the stage of maximum inverting activity. Then add neutral lead acetate in slight excess, filter, precipitate the lead in the filtrate with hydrogen sulphid, filter again and then dialyze the filtrate thoroughly in a collodion sac. Preserve in an ice box the dialyzed solution with the addition of a little toluene to prevent the growth of micro-organisms. Note the optical activity of the invertase solution and correct the invert reading according to the amount of the solution used.

16

DETERMINATION.

Dissolve the normal weight (26 grams) of the substance in water, clarify, make up to volume, and take the direct polarization (P) as directed under 14. If lead has been used as a clarifying agent, remove the excess of lead from the filtrate, with anhydrous sodium carbonate or potassium oxalate, and filter. To 50 cc. of the filtrate in a 100 cc. flask add acetic acid, drop by drop, until the reaction is acid to litmus, add 10 cc. of the invertase solution, fill the flask with water nearly to 100 cc. and let stand in a warm place (about 40°C.) overnight. Cool and make up to 100 cc. at 20°C. Polarize at 20°C. in a 200 mm. tube. Allow the solution to remain in the tube for an hour and repeat the polarization. If there is no change from the previous reading, the inversion is complete, whereupon the reading and temperature of the solution are carefully noted. Correct the reading for the optical activity of the invertase solution and then multiply by 2. Calculate the percentage of sucrose by the following formula:

$$S = \frac{100 (P - I)}{142 - \frac{T}{2} - 0.0065 \left[142 - \frac{T}{2} - (P - I) \right]} \text{ in which}$$

S = per cent of sucrose;
 P = direct reading;
 I = invert reading;
 T = temperature at which invert reading is made.

17

DETERMINATION OF SUCROSE AND RAFFINOSE.—OFFICIAL.

(Of value chiefly in the analysis of beet products.)

If the direct reading is more than 1° higher than the per cent of sucrose as calculated by the formula given under 14, raffinose is probably present. Calculate sucrose and raffinose by the following formula of Hersfeld:

$$S = \frac{0.5124 P - I}{0.839}; \quad R = \frac{P - S}{1.852} \text{ in which}$$

P = direct reading normal solution;

I = invert reading normal solution;

S = per cent of sucrose;

R = per cent of anhydrous raffinose.

The above formula assumes that the polarisations are made at exactly 20°C. If the temperature (T) is other than 20°C., the following formula should be used:

$$S = \frac{P (0.4724 + 0.002 T) - I}{0.899 - 0.003 T}$$

Having calculated S, then $R = \frac{P - S}{1.852}$

CHEMICAL METHODS.

18 DETERMINATION OF SUCROSE FROM REDUCING SUGARS BEFORE AND AFTER INVERSION.—TENTATIVE.

Determine the reducing sugars (clarification having been effected with *neutral* lead acetate, never with basic lead acetate), as directed under 25, and calculate to invert sugar from 27. Invert the solution as directed under 14 or 16, exactly neutralize the acid, and again determine the reducing sugars, but calculate them to invert sugar from the same table as referred to above, using the invert sugar column alone. Deduct the percentage of invert sugar obtained before inversion from that obtained after inversion, and multiply the difference by 0.95, the result being the per cent of sucrose. The solutions should be diluted in both determinations so that not more than 245 mg. of invert sugar are present in the amount taken for reduction. It is important that all lead be removed from the solution with potassium oxalate before reduction.

REDUCING SUGARS.

INVERT SUGAR.

Approximate Volumetric Method for Rapid Work.—Tentative.

19

REAGENT.

Soxhlet's Modification of Fehling's Solution.—Prepare by mixing, immediately before use, equal volumes of (a) and (b).

(a) *Copper sulphate solution.*—Dissolve 34.639 grams of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water, dilute to 500 cc. and filter through prepared asbestos.

(b) *Alkaline tartrate solution.*—Dissolve 173 grams of Rochelle salts and 50 grams of sodium hydroxid in water, dilute to 500 cc., allow to stand for 2 days and filter through prepared asbestos.

20

STANDARDIZATION OF COPPER SOLUTION.

Since the factor of calculation varies with the minute details of manipulation, every operator must determine a factor for himself, using a known solution of the pure sugar that he desires to determine, and keeping the conditions the same as those used for the determination.

Standardize the solution for invert sugar in the following manner:

Dissolve 4.75 grams of pure sucrose in 75 cc. of water, add 5 cc. of hydrochloric acid (sp. gr. 1.20) and invert as directed under 14. Neutralize the acid with sodium hydroxid solution and dilute to 1 liter. Ten cc. of this solution contain 0.050 gram of invert sugar, which should reduce 10 cc. of the reagent. The strength of the copper solution should never be taken as a constant, but should be checked against the sugar.

21

DETERMINATION.

Place 10 cc. of the reagent in a large test tube and add 10 cc. of water. Heat to boiling, and add gradually small portions of the solution of the material to be tested until the copper has been completely reduced, boiling after each addition to complete the reaction. Two minutes' boiling is required for complete reduction when the full amount of sugar solution has been added in one portion. When the end is nearly reached and the amount of sugar solution to be added can no longer be judged by the color of the solution, remove a small portion of the liquid and filter rapidly into a small porcelain crucible or on a test plate; acidify with dilute acetic acid, and test for copper with dilute potassium ferrocyanid solution. The sugar solution should be of such strength as will give a burette reading of 15–20 cc., and the number of successive additions should be as small as possible.

Soxhlet Volumetric Method.—Tentative.

22

REAGENT.

The reagent used is described under 19.

23

DETERMINATION.

Make a preliminary titration to determine the approximate percentage of reducing sugar in the material under examination. Prepare a solution which contains approximately 1% of reducing sugar. Place in a beaker 100 cc. of the reagent and approximately the amount of the sugar solution for its complete reduction. Boil for 2 minutes. Filter through a folded filter and test a portion of the filtrate for copper by use of dilute acetic acid and dilute potassium ferrocyanid solution. Repeat, varying the volume of sugar solution, until 2 successive amounts are found which differ by 0.1 cc., one giving complete reduction and the other leaving a small amount of copper in solution. The mean of these 2 readings is taken as the volume of the solution required for the complete precipitation of 100 cc. of the reagent.

Under these conditions 100 cc. of the reagent require 0.494 gram of invert sugar for complete reduction. Calculate the percentage by the following formula:

V = the volume of the sugar solution required for the complete reduction of 100 cc. of the reagent;

W = the weight of the sample in 1 cc. of the sugar solution;

$\frac{100 \times 0.494}{VW}$ = per cent of invert sugar.

GRAVIMETRIC METHODS.

Munson and Walker General Method.²—Tentative.

24

REAGENTS.

(a) *Asbestos*.—Digest the asbestos, which should be the amphibole variety, with dilute hydrochloric acid (1 to 3) for 2–3 days. Wash free from acid, digest for a

similar period with 10% sodium hydroxid solution, and then treat for a few hours with hot alkaline tartrate solution (old alkaline tartrate solutions that have stood for some time may be used for this purpose) of the strength employed in sugar determinations. Then wash the asbestos free from alkali, digest for several hours with dilute nitric acid (1 to 3) and, after washing free from acid, shake with water into a fine pulp. In preparing the Gooch crucible, make a film of asbestos $\frac{1}{2}$ inch thick and wash thoroughly with water to remove fine particles of asbestos. If the precipitated cuprous oxid is to be weighed as such, wash the crucible with 10 cc. of alcohol, then with 10 cc. of ether, dry for 30 minutes at 100°C., cool in a desiccator and weigh.

(b) The solution used is described under 19.

25**PRECIPITATION OF CUPROUS OXID.**

Transfer 25 cc. each of the copper sulphate and alkaline tartrate solutions to a 400 cc. beaker of alkali-resisting glass and add 50 cc. of reducing sugar solution, or, if a smaller volume of sugar solution is used, add water to make the final volume 100 cc. Heat the beaker upon an asbestos gauze over a Bunsen burner, regulate the flame so that boiling begins in 4 minutes, and continue the boiling for exactly 2 minutes. (It is important that these directions be strictly observed and, in order to regulate the burner for this purpose, it is advisable to make preliminary tests, using 50 cc. of the reagent and 50 cc. of water before proceeding with the actual determination.) Keep the beaker covered with a watch glass during the heating. Filter the cuprous oxid at once on an asbestos mat in a porcelain Gooch crucible, using suction. Wash the cuprous oxid thoroughly with water at a temperature of about 60°C., and either weigh directly as cuprous oxid as in 26, or, determine the amount of reduced copper by one of the methods under 29-34, respectively. Conduct a blank determination, using 50 cc. of the reagent and 50 cc. of water, and, if the weight of cuprous oxid obtained exceeds 0.5 mg., correct the result of the reducing sugar determination accordingly. The alkaline tartrate solution deteriorates on standing and the amount of cuprous oxid obtained in the blank increases.

DETERMINATION OF REDUCED COPPER.**26*****I. Direct Weighing of Cuprous Oxid.—Tentative.***

Prepare a Gooch as directed under 24 (a).

Collect the precipitated cuprous oxid on the mat, as directed under 25, wash thoroughly with hot water, then with 10 cc. of alcohol, and finally with 10 cc. of ether. Dry the precipitate for 30 minutes in a water oven at the temperature of boiling water; cool and weigh. Calculate the weight of metallic copper. Obtain from 27 the weight of invert sugar equivalent to the weight of copper found.

This method should be used only for determinations in pure sugar solutions. In all other products the copper of the cuprous oxid should be determined by one of the following methods, since the cuprous oxid is very apt to be contaminated with foreign matter.

The number of milligrams of copper reduced by a given amount of reducing sugar differs when sucrose is present and when it is absent. In the tables the absence of sucrose is assumed except in the two columns under invert sugar, where one for mixtures of invert sugar and sucrose containing 0.4 gram of total sugar in 50 cc. of solution, and one for invert sugar and sucrose when the 50 cc. of solution contains 2 grams of total sugar are given, in addition to the column for invert sugar alone.

27

TABLE 1.—MUNSON AND WALKER'S TABLE.

For calculating dextrose, invert sugar alone, invert sugar in the presence of sucrose (0.4 gram and 2 grams total sugar), lactose (two forms), and mallose (anhydrous and crystallized).

[Expressed in milligrams.]

CURCUMOUS OXID (Cu ₂ O)	COPPER (Cu)	DEXTRINE (d-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CURCUMOUS OXID (Cu ₂ O)
				0.4 gram total sugar	2 grams total sugar	Cu ₂ H ₂ O ₁₁	Cu ₂ H ₂ O ₁₁ H ₂ O	Cu ₂ H ₂ O ₁₁	Cu ₂ H ₂ O ₁₁ H ₂ O	
10	8.9	4.0	4.5	1.6	3.8	4.0	5.9	6.2	10
11	9.8	4.5	5.0	2.1	4.5	4.7	6.7	7.0	11
12	10.7	4.9	5.4	2.5	5.1	5.4	7.5	7.9	12
13	11.5	5.3	5.8	3.0	5.8	6.1	8.3	8.7	13
14	12.4	5.7	6.3	3.4	6.4	6.8	9.1	9.5	14
15	13.3	6.2	6.7	3.9	7.1	7.5	9.9	10.4	15
16	14.2	6.6	7.2	4.3	7.8	8.2	10.6	11.2	16
17	15.1	7.0	7.6	4.8	8.4	8.9	11.4	12.0	17
18	16.0	7.5	8.1	5.2	9.1	9.5	12.2	12.9	18
19	16.9	7.9	8.5	5.7	9.7	10.2	13.0	13.7	19
20	17.8	8.3	8.9	6.1	10.4	10.9	13.8	14.6	20
21	18.7	8.7	9.4	6.6	11.0	11.6	14.6	15.4	21
22	19.5	9.2	9.8	7.0	11.7	12.3	15.4	16.2	22
23	20.4	9.6	10.3	7.5	12.3	13.0	16.2	17.1	23
24	21.3	10.0	10.7	7.9	13.0	13.7	17.0	17.9	24
25	22.2	10.5	11.2	8.4	13.7	14.4	17.8	18.7	25
26	23.1	10.9	11.6	8.8	14.3	15.1	18.6	19.6	26
27	24.0	11.3	12.0	9.3	15.0	15.8	19.4	20.4	27
28	24.9	11.8	12.5	9.7	15.6	16.5	20.2	21.2	28
29	25.8	12.2	12.9	10.2	16.3	17.1	21.0	22.1	29
30	26.6	12.6	13.4	10.7	4.3	16.9	17.8	21.8	22.9	30
31	27.5	13.1	13.8	11.1	4.7	17.6	18.5	22.6	23.7	31
32	28.4	13.5	14.3	11.6	5.2	18.3	19.2	23.3	24.6	32
33	29.3	13.9	14.7	12.0	5.6	18.9	19.9	24.1	25.4	33
34	30.2	14.3	15.2	12.5	6.1	19.6	20.6	24.9	26.2	34
35	31.1	14.8	15.6	12.9	6.5	20.2	21.3	25.7	27.1	35
36	32.0	15.2	16.1	13.4	7.0	20.9	22.0	26.5	27.9	36
37	32.9	15.6	16.5	13.8	7.4	21.5	22.7	27.3	28.7	37
38	33.8	16.1	16.9	14.3	7.9	22.2	23.4	28.1	29.6	38
39	34.6	16.5	17.4	14.7	8.4	22.8	24.1	28.9	30.4	39
40	35.5	16.9	17.8	15.2	8.8	23.5	24.8	29.7	31.3	40
41	36.4	17.4	18.3	15.6	9.3	24.2	25.4	30.5	32.1	41
42	37.3	17.8	18.7	16.1	9.7	24.8	26.1	31.3	32.9	42
43	38.2	18.2	19.2	16.6	10.2	25.5	26.8	32.1	33.8	43
44	39.1	18.7	19.6	17.0	10.7	26.1	27.5	32.9	34.6	44
45	40.0	19.1	20.1	17.5	11.1	26.8	28.2	33.7	35.4	45
46	40.9	19.6	20.5	17.9	11.6	27.4	28.9	34.4	36.3	46
47	41.7	20.0	21.0	18.4	12.0	28.1	29.6	35.2	37.1	47
48	42.6	20.4	21.4	18.8	12.5	28.7	30.3	36.0	37.9	48
49	43.5	20.9	21.9	19.3	12.9	29.4	31.0	36.8	38.8	49
50	44.4	21.3	22.3	19.7	13.4	30.1	31.7	37.6	39.6	50
51	45.3	21.7	22.8	20.2	13.9	30.7	32.4	38.4	40.4	51
52	46.2	22.2	23.2	20.7	14.3	31.4	33.0	39.2	41.3	52
53	47.1	22.6	23.7	21.1	14.8	32.1	33.7	40.0	42.1	53
54	48.0	23.0	24.1	21.6	15.2	32.7	34.4	40.8	42.9	54
55	48.9	23.5	24.6	22.0	15.7	33.4	35.1	41.6	43.8	55
56	49.7	23.9	25.0	22.5	16.2	34.0	35.8	42.4	44.6	56
57	50.6	24.3	25.5	22.9	16.6	34.7	36.5	43.2	45.4	57
58	51.5	24.8	25.9	23.4	17.1	35.4	37.2	44.0	46.3	58
59	52.4	25.2	26.4	23.9	17.5	36.0	37.9	44.8	47.1	59
60	53.3	25.6	26.8	24.3	18.0	36.7	38.6	45.6	48.0	60
61	54.2	26.1	27.3	24.8	18.5	37.3	39.3	46.3	48.8	61
62	55.1	26.5	27.7	25.2	18.9	38.0	40.0	47.1	49.6	62
63	56.0	27.0	28.2	25.7	19.4	38.6	40.7	47.9	50.5	63
64	56.8	27.4	28.6	26.2	19.8	39.3	41.4	48.7	51.3	64

27

TABLE 1.—MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu ₂ O)	COPPER (Cu)	DEXTRINE (d-glucose)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu ₂ O)
				0.4 gram total sugar	2 grams total sugar	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ ·H ₂ O	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ ·H ₂ O	
65	57.7	27.8	29.1	26.6	20.3	40.0	42.1	49.5	52.1	65
66	58.6	28.3	29.5	27.1	20.8	40.6	42.8	50.3	53.0	66
67	59.5	28.7	30.0	27.6	21.2	41.3	43.6	51.1	53.8	67
68	60.4	29.2	30.4	28.0	21.7	41.9	44.2	51.9	54.6	68
69	61.3	29.6	30.9	28.5	22.2	42.6	44.8	52.7	55.5	69
70	62.2	30.0	31.3	28.9	22.6	43.3	45.5	53.5	56.3	70
71	63.1	30.5	31.8	29.4	23.1	43.9	46.2	54.3	57.1	71
72	64.0	30.9	32.3	29.8	23.5	44.6	46.9	55.1	58.0	72
73	64.8	31.4	32.7	30.3	24.0	45.2	47.6	55.9	58.8	73
74	65.7	31.8	33.2	30.8	24.5	45.9	48.3	56.7	59.6	74
75	66.6	32.2	33.6	31.2	24.9	46.6	49.0	57.5	60.5	75
76	67.5	32.7	34.1	31.7	25.4	47.2	49.7	58.2	61.3	76
77	68.4	33.1	34.5	32.1	25.9	47.9	50.4	59.0	62.1	77
78	69.3	33.6	35.0	32.6	26.3	48.5	51.1	59.8	63.0	78
79	70.2	34.0	35.4	33.1	26.8	49.2	51.8	60.6	63.8	79
80	71.1	34.4	35.9	33.5	27.3	49.9	52.5	61.4	64.6	80
81	71.9	34.9	36.3	34.0	27.7	50.5	53.2	62.2	65.5	81
82	72.8	35.3	36.8	34.5	28.2	51.2	53.9	63.0	66.3	82
83	73.7	35.8	37.3	34.9	28.6	51.8	54.6	63.8	67.1	83
84	74.6	36.2	37.7	35.4	29.1	52.5	55.3	64.6	68.0	84
85	75.5	36.7	38.2	35.8	29.6	53.1	56.0	65.4	68.8	85
86	76.4	37.1	38.6	36.3	30.0	53.8	56.6	66.2	69.7	86
87	77.3	37.5	39.1	36.8	30.5	54.5	57.3	67.0	70.5	87
88	78.2	38.0	39.5	37.2	31.0	55.1	58.0	67.8	71.3	88
89	79.1	38.4	40.0	37.7	31.4	55.8	58.7	68.5	72.2	89
90	79.9	38.9	40.4	38.2	31.9	56.4	59.4	69.3	73.0	90
91	80.8	39.3	40.9	38.6	32.4	57.1	60.1	70.1	73.8	91
92	81.7	39.8	41.4	39.1	32.8	57.8	60.8	70.9	74.7	92
93	82.6	40.2	41.8	39.6	33.3	58.4	61.5	71.7	75.5	93
94	83.5	40.6	42.3	40.0	33.8	59.1	62.2	72.5	76.3	94
95	84.4	41.1	42.7	40.5	34.2	59.7	62.9	73.3	77.2	95
96	85.3	41.5	43.2	41.0	34.7	60.4	63.6	74.1	78.0	96
97	86.2	42.0	43.7	41.4	35.2	61.1	64.3	74.9	78.8	97
98	87.1	42.4	44.1	41.9	35.6	61.7	65.0	75.7	79.7	98
99	87.9	42.9	44.6	42.4	36.1	62.4	65.7	76.5	80.5	99
100	88.8	43.3	45.0	42.8	36.6	63.0	66.4	77.3	81.3	100
101	89.7	43.8	45.5	43.3	37.0	63.7	67.1	78.1	82.2	101
102	90.6	44.2	46.0	43.8	37.5	64.4	67.8	78.8	83.0	102
103	91.5	44.7	46.4	44.2	38.0	65.0	68.5	79.6	83.8	103
104	92.4	45.1	46.9	44.7	38.5	65.7	69.1	80.4	84.7	104
105	93.3	45.5	47.3	45.2	38.9	66.4	69.8	81.2	85.5	105
106	94.2	46.0	47.8	45.6	39.4	67.0	70.5	82.0	86.3	106
107	95.0	46.4	48.3	46.1	39.9	67.7	71.2	82.8	87.2	107
108	95.9	46.9	48.7	46.6	40.3	68.3	71.9	83.6	88.0	108
109	96.8	47.3	49.2	47.0	40.8	69.0	72.6	84.4	88.8	109
110	97.7	47.8	49.6	47.5	41.3	69.7	73.3	85.2	89.7	110
111	98.6	48.2	50.1	48.0	41.7	70.3	74.0	86.0	90.5	111
112	99.5	48.7	50.6	48.4	42.2	71.0	74.7	86.8	91.3	112
113	100.4	49.1	51.0	48.9	42.7	71.6	75.4	87.6	92.2	113
114	101.3	49.6	51.5	49.4	43.2	72.3	76.1	88.4	93.0	114
115	102.2	50.0	51.9	49.8	43.6	73.0	76.8	89.2	93.9	115
116	103.0	50.5	52.4	50.3	44.1	73.6	77.5	90.0	94.7	116
117	103.9	50.9	52.9	50.8	44.6	74.3	78.2	90.7	95.5	117
118	104.8	51.4	53.3	51.2	45.0	75.0	78.9	91.5	96.4	118
119	105.7	51.8	53.8	51.7	45.5	75.6	79.6	92.3	97.2	119

TABLE 1.—MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu ₂ O)	COPPER (Cu)	DEXTROSE (d-glucose)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu ₂ O)
				0.4 gram sugar	2 grams sugar	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	
120	106.6	52.8	54.3	52.2	46.0	76.3	80.3	93.1	96.0	120
121	107.5	52.7	54.7	52.7	46.5	76.9	81.0	93.9	96.9	121
122	108.4	53.2	55.2	53.1	46.9	77.6	81.7	94.7	99.7	122
123	109.3	53.6	55.7	53.6	47.4	78.3	82.4	95.5	100.5	123
124	110.1	54.1	56.1	54.1	47.9	78.9	83.1	96.3	101.4	124
125	111.0	54.5	56.6	54.5	48.3	79.6	83.8	97.1	102.2	125
126	111.9	55.0	57.0	55.0	48.8	80.3	84.5	97.9	103.0	126
127	112.8	55.4	57.5	55.5	49.3	80.9	85.2	98.7	103.9	127
128	113.7	55.9	58.0	55.9	49.8	81.6	85.9	99.4	104.7	128
129	114.6	56.3	58.4	56.4	50.2	82.2	86.6	100.2	105.5	129
130	115.5	56.8	58.9	56.9	50.7	82.9	87.3	101.0	106.4	130
131	116.4	57.2	59.4	57.4	51.2	83.6	88.0	101.8	107.2	131
132	117.3	57.7	59.8	57.8	51.7	84.2	88.7	102.6	108.0	132
133	118.1	58.1	60.3	58.3	52.1	84.9	89.4	103.4	108.9	133
134	119.0	58.6	60.8	58.8	52.6	85.5	90.1	104.2	109.7	134
135	119.9	59.0	61.2	59.3	53.1	86.2	90.8	105.0	110.5	135
136	120.8	59.5	61.7	59.7	53.6	86.9	91.5	105.8	111.4	136
137	121.7	60.0	62.2	60.2	54.0	87.5	92.1	106.6	112.2	137
138	122.6	60.4	62.6	60.7	54.5	88.2	92.8	107.4	113.0	138
139	123.5	60.9	63.1	61.2	55.0	88.9	93.5	108.2	113.9	139
140	124.4	61.3	63.6	61.6	55.5	89.5	94.2	109.0	114.7	140
141	125.2	61.8	64.0	62.1	55.9	90.2	94.9	109.8	115.5	141
142	126.1	62.2	64.5	62.6	56.4	90.8	95.6	110.5	116.4	142
143	127.0	62.7	65.0	63.1	56.9	91.5	96.3	111.3	117.2	143
144	127.9	63.1	65.4	63.5	57.4	92.2	97.0	112.1	118.0	144
145	128.8	63.6	65.9	64.0	57.8	92.8	97.7	112.9	118.9	145
146	129.7	64.0	66.4	64.5	58.3	93.5	98.4	113.7	119.7	146
147	130.6	64.5	66.9	65.0	58.8	94.2	99.1	114.5	120.5	147
148	131.5	65.0	67.3	65.4	59.3	94.8	99.8	115.3	121.4	148
149	132.4	65.4	67.8	65.9	59.7	95.5	100.5	116.1	122.2	149
150	133.2	65.9	68.2	66.4	60.2	96.1	101.2	116.9	123.0	150
151	134.1	66.3	68.7	66.9	60.7	96.8	101.9	117.7	123.9	151
152	135.0	66.8	69.2	67.3	61.2	97.5	102.6	118.5	124.7	152
153	135.9	67.2	69.7	67.8	61.7	98.1	103.3	119.3	125.5	153
154	136.8	67.7	70.1	68.3	62.1	98.8	104.0	120.0	126.4	154
155	137.7	68.2	70.6	68.8	62.6	99.5	104.7	120.8	127.2	155
156	138.6	68.6	71.1	69.2	63.1	100.1	105.4	121.6	128.0	156
157	139.5	69.1	71.6	69.7	63.6	100.8	106.1	122.4	128.9	157
158	140.3	69.5	72.0	70.2	64.1	101.5	106.8	123.2	129.7	158
159	141.2	70.0	72.5	70.7	64.5	102.1	107.5	124.0	130.5	159
160	142.1	70.4	73.0	71.2	65.0	102.8	108.2	124.8	131.4	160
161	143.0	70.9	73.4	71.6	65.5	103.4	108.9	125.6	132.2	161
162	143.9	71.4	73.9	72.1	66.0	104.1	109.6	126.4	133.0	162
163	144.8	71.8	74.4	72.6	66.5	104.8	110.3	127.2	133.9	163
164	145.7	72.3	74.9	73.1	66.9	105.4	111.0	128.0	134.7	164
165	146.6	72.8	75.3	73.6	67.4	106.1	111.7	128.8	135.5	165
166	147.5	73.2	75.8	74.0	67.9	106.8	112.4	129.6	136.4	166
167	148.3	73.7	76.3	74.5	68.4	107.4	113.1	130.3	137.2	167
168	149.2	74.1	76.8	75.0	68.9	108.1	113.8	131.1	138.0	168
169	150.1	74.6	77.2	75.5	69.3	108.8	114.5	131.9	138.9	169
170	151.0	75.1	77.7	76.0	69.8	109.4	115.2	132.7	139.7	170
171	151.9	75.5	78.2	76.4	70.3	110.1	115.9	133.5	140.5	171
172	152.8	76.0	78.7	76.9	70.8	110.8	116.6	134.3	141.4	172
173	153.7	76.4	79.1	77.4	71.3	111.4	117.3	135.1	142.2	173
174	154.6	76.9	79.6	77.9	71.7	112.1	118.0	135.9	143.0	174

TABLE 1.—MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPREOUS OXID (CuO)	COPPER (Cu)	DEXTROSE (d-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPREOUS OXID (CuO)
				0.4 gram sugar	2 grams sugar	CuH ₁₂ O ₁₁	CuH ₁₂ O ₁₁ H ₂ O	CuH ₁₂ O ₁₁	CuH ₁₂ O ₁₁ H ₂ O	
175	155.5	77.4	80.1	78.4	72.3	112.8	118.7	136.7	143.9	175
176	156.3	77.8	80.6	78.8	72.7	113.4	119.4	137.5	144.7	176
177	157.2	78.3	81.0	79.3	73.2	114.1	120.1	138.3	145.5	177
178	158.1	78.8	81.5	79.8	73.7	114.8	120.8	139.1	146.4	178
179	159.0	79.2	82.0	80.3	74.2	115.4	121.5	139.8	147.2	179
180	159.9	79.7	82.5	80.8	74.6	116.1	122.2	140.6	148.0	180
181	160.8	80.1	82.9	81.3	75.1	116.7	122.9	141.4	148.9	181
182	161.7	80.6	83.4	81.7	75.6	117.4	123.6	142.2	149.7	182
183	162.6	81.1	83.9	82.2	76.1	118.1	124.3	143.0	150.5	183
184	163.4	81.5	84.4	82.7	76.6	118.7	125.0	143.8	151.4	184
185	164.3	82.0	84.9	83.2	77.1	119.4	125.7	144.6	152.2	185
186	165.2	82.5	85.3	83.7	77.6	120.1	126.4	145.4	153.0	186
187	166.1	82.9	85.8	84.2	78.0	120.7	127.1	146.2	153.9	187
188	167.0	83.4	86.3	84.6	78.5	121.4	127.8	147.0	154.7	188
189	167.9	83.9	86.8	85.1	79.0	122.1	128.5	147.8	155.5	189
190	168.8	84.3	87.2	85.6	79.5	122.7	129.2	148.6	156.4	190
191	169.7	84.8	87.7	86.1	80.0	123.4	129.9	149.3	157.2	191
192	170.5	85.3	88.2	86.6	80.5	124.1	130.6	150.1	158.0	192
193	171.4	85.7	88.7	87.1	81.0	124.7	131.3	150.9	158.9	193
194	172.3	86.2	89.2	87.6	81.4	125.4	132.0	151.7	159.7	194
195	173.2	86.7	89.6	88.0	81.9	126.1	132.7	152.5	160.5	195
196	174.1	87.1	90.1	88.5	82.4	126.7	133.4	153.3	161.4	196
197	175.0	87.6	90.6	89.0	82.9	127.4	134.1	154.1	162.2	197
198	175.9	88.1	91.1	89.5	83.4	128.1	134.8	154.9	163.0	198
199	176.8	88.5	91.6	90.0	83.9	128.7	135.5	155.7	163.9	199
200	177.7	89.0	92.0	90.5	84.4	129.4	136.2	156.5	164.7	200
201	178.5	89.5	92.5	91.0	84.8	130.0	136.9	157.3	165.5	201
202	179.4	89.9	93.0	91.4	85.3	130.7	137.6	158.1	166.4	202
203	180.3	90.4	93.5	91.9	85.8	131.4	138.3	158.8	167.2	203
204	181.2	90.9	94.0	92.4	86.3	132.0	139.0	159.6	168.0	204
205	182.1	91.4	94.5	92.9	86.8	132.7	139.7	160.4	168.9	205
206	183.0	91.8	94.9	93.4	87.3	133.4	140.4	161.2	169.7	206
207	183.9	92.3	95.4	93.9	87.8	134.0	141.1	162.0	170.5	207
208	184.8	92.8	95.9	94.4	88.3	134.7	141.8	162.8	171.4	208
209	185.6	93.2	96.4	94.9	88.8	135.4	142.5	163.6	172.2	209
210	186.5	93.7	96.9	95.4	89.2	136.0	143.2	164.4	173.0	210
211	187.4	94.2	97.4	95.8	89.7	136.7	143.9	165.2	173.8	211
212	188.3	94.6	97.8	96.3	90.2	137.4	144.6	166.0	174.7	212
213	189.2	95.1	98.3	96.8	90.7	138.0	145.3	166.8	175.5	213
214	190.1	95.6	98.8	97.3	91.2	138.7	146.0	167.6	176.4	214
215	191.0	96.1	99.3	97.8	91.7	139.4	146.7	168.3	177.2	215
216	191.9	96.5	99.8	98.3	92.2	140.0	147.4	169.1	178.0	216
217	192.8	97.0	100.3	98.8	92.7	140.7	148.1	169.9	178.9	217
218	193.6	97.5	100.8	99.3	93.2	141.4	148.8	170.7	179.7	218
219	194.5	98.0	101.2	99.8	93.7	142.0	149.5	171.5	180.5	219
220	195.4	98.4	101.7	100.3	94.2	142.7	150.2	172.3	181.4	220
221	196.3	98.9	102.2	100.8	94.7	143.4	150.9	173.1	182.2	221
222	197.2	99.4	102.7	101.3	95.1	144.0	151.6	173.9	183.0	222
223	198.1	99.9	103.2	101.7	95.6	144.7	152.3	174.7	183.9	223
224	199.0	100.3	103.7	102.2	96.1	145.4	153.0	175.5	184.7	224
225	199.9	100.8	104.2	102.7	96.6	146.0	153.7	176.2	185.5	225
226	200.7	101.3	104.6	103.2	97.1	146.7	154.4	177.0	186.4	226
227	201.6	101.8	105.1	103.7	97.6	147.4	155.1	177.8	187.2	227
228	202.5	102.2	105.6	104.2	98.1	148.0	155.8	178.6	188.0	228
229	203.4	102.7	106.1	104.7	98.6	148.7	156.5	179.4	188.8	229

TABLE 1.—MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu ₂ O)	COPPER (Cu)	DEXTROSE (d-glucose)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu ₂ O)
				0.4 gram sugar	2 grams sugar	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ ·H ₂ O	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ ·H ₂ O	
230	204.3	103.2	106.6	105.2	99.1	149.4	157.2	180.2	189.7	230
231	205.2	103.7	107.1	105.7	99.6	150.0	157.9	181.0	190.5	231
232	206.1	104.1	107.6	106.2	100.1	150.7	158.6	181.8	191.3	232
233	207.0	104.6	108.1	106.7	100.6	151.4	159.3	182.6	192.2	233
234	207.9	105.1	108.6	107.2	101.1	152.0	160.0	183.4	193.0	234
235	208.7	105.6	109.1	107.7	101.6	152.7	160.7	184.2	193.8	235
236	209.6	106.0	109.5	108.2	102.1	153.4	161.4	184.9	194.7	236
237	210.5	106.5	110.0	108.7	102.6	154.0	162.1	185.7	195.5	237
238	211.4	107.0	110.5	109.2	103.1	154.7	162.8	186.5	196.3	238
239	212.3	107.5	111.0	109.6	103.5	155.4	163.5	187.3	197.2	239
240	213.2	108.0	111.5	110.1	104.0	156.1	164.3	188.1	198.0	240
241	214.1	108.4	112.0	110.6	104.5	156.7	165.0	188.9	198.8	241
242	215.0	108.9	112.5	111.1	105.0	157.4	165.7	189.7	199.7	242
243	215.8	109.4	113.0	111.6	105.5	158.1	166.4	190.5	200.5	243
244	216.7	109.9	113.5	112.1	106.0	158.7	167.1	191.3	201.3	244
245	217.6	110.4	114.0	112.6	106.5	159.4	167.8	192.1	202.2	245
246	218.5	110.8	114.5	113.1	107.0	160.1	168.5	192.9	203.0	246
247	219.4	111.3	115.0	113.6	107.5	160.7	169.2	193.6	203.8	247
248	220.3	111.8	115.4	114.1	108.0	161.4	169.9	194.4	204.7	248
249	221.2	112.3	115.9	114.6	108.5	162.1	170.6	195.2	205.5	249
250	222.1	112.8	116.4	115.1	109.0	162.7	171.3	196.0	206.3	250
251	223.0	113.2	116.9	115.6	109.5	163.4	172.0	196.8	207.2	251
252	223.8	113.7	117.4	116.1	110.0	164.1	172.7	197.6	208.0	252
253	224.7	114.2	117.9	116.6	110.5	164.7	173.4	198.4	208.8	253
254	225.6	114.7	118.4	117.1	111.0	165.4	174.1	199.2	209.7	254
255	226.5	115.2	118.9	117.6	111.5	166.1	174.8	200.0	210.5	255
256	227.4	115.7	119.4	118.1	112.0	166.8	175.5	200.8	211.3	256
257	228.3	116.1	119.9	118.6	112.5	167.4	176.2	201.6	212.2	257
258	229.2	116.6	120.4	119.1	113.0	168.1	176.9	202.3	213.0	258
259	230.1	117.1	120.9	119.6	113.5	168.8	177.6	203.1	213.8	259
260	231.0	117.6	121.4	120.1	114.0	169.4	178.3	203.9	214.7	260
261	231.8	118.1	121.9	120.6	114.5	170.1	179.0	204.7	215.5	261
262	232.7	118.6	122.4	121.1	115.0	170.8	179.8	205.5	216.3	262
263	233.6	119.0	122.9	121.6	115.5	171.4	180.5	206.3	217.2	263
264	234.5	119.5	123.4	122.1	116.0	172.1	181.2	207.1	218.0	264
265	235.4	120.0	123.9	122.6	116.5	172.8	181.9	207.9	218.8	265
266	236.3	120.5	124.4	123.1	117.0	173.5	182.6	208.7	219.7	266
267	237.2	121.0	124.9	123.6	117.5	174.1	183.3	209.5	220.5	267
268	238.1	121.5	125.4	124.1	118.0	174.8	184.0	210.3	221.3	268
269	238.9	122.0	125.9	124.6	118.5	175.5	184.7	211.0	222.1	269
270	239.8	122.5	126.4	125.1	119.0	176.1	185.4	211.8	223.0	270
271	240.7	122.9	126.9	125.6	119.5	176.8	186.1	212.6	223.8	271
272	241.6	123.4	127.4	126.2	120.0	177.5	186.8	213.4	224.6	272
273	242.5	123.9	127.9	126.7	120.6	178.1	187.5	214.2	225.5	273
274	243.4	124.4	128.4	127.2	121.1	178.8	188.2	215.0	226.3	274
275	244.3	124.9	128.9	127.7	121.6	179.5	188.9	215.8	227.1	275
276	245.2	125.4	129.4	128.2	122.1	180.2	189.6	216.6	228.0	276
277	246.1	125.9	129.9	128.7	122.6	180.8	190.3	217.4	228.8	277
278	246.9	126.4	130.4	129.2	123.1	181.5	191.0	218.2	229.6	278
279	247.8	126.9	130.9	129.7	123.6	182.2	191.7	218.9	230.5	279
280	248.7	127.3	131.4	130.2	124.1	182.8	192.4	219.7	231.3	280
281	249.6	127.8	131.9	130.7	124.6	183.5	193.1	220.5	232.1	281
282	250.5	128.3	132.4	131.2	125.1	184.2	193.9	221.3	233.0	282
283	251.4	128.8	132.9	131.7	125.6	184.8	194.6	222.1	233.8	283
284	252.3	129.3	133.4	132.2	126.1	185.5	195.3	222.9	234.6	284

TABLE 1.—MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu ₂ O)	COPPER (Cu)	DEXTRINE (d-glucose)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu ₂ O)
				0.4 gram sugar	2 grams sugar	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	
285	253.2	129.8	133.9	132.7	126.6	186.2	196.0	223.7	235.5	285
286	254.0	130.3	134.4	133.2	127.1	186.9	196.7	224.5	236.3	286
287	254.9	130.8	134.9	133.7	127.6	187.5	197.4	225.3	237.1	287
288	255.8	131.3	135.4	134.3	128.1	188.2	198.1	226.1	238.0	288
289	256.7	131.8	135.9	134.8	128.6	188.9	198.8	226.9	238.8	289
290	257.6	132.3	136.4	135.3	129.2	189.5	199.5	227.6	239.6	290
291	258.5	132.7	136.9	135.8	129.7	190.2	200.2	228.4	240.5	291
292	259.4	133.2	137.4	136.3	130.2	190.9	200.9	229.2	241.3	292
293	260.3	133.7	137.9	136.8	130.7	191.5	201.6	230.0	242.1	293
294	261.2	134.2	138.4	137.3	131.2	192.2	202.3	230.8	242.9	294
295	262.0	134.7	138.9	137.8	131.7	192.9	203.0	231.6	243.8	295
296	262.9	135.2	139.4	138.3	132.2	193.6	203.7	232.4	244.6	296
297	263.8	135.7	140.0	139.8	132.7	194.2	204.4	233.2	245.4	297
298	264.7	136.2	140.5	139.4	133.2	194.9	205.1	234.0	246.3	298
299	265.6	136.7	141.0	139.9	133.7	195.6	205.8	234.8	247.1	299
300	266.5	137.2	141.5	140.4	134.2	196.2	206.6	235.5	247.9	300
301	267.4	137.7	142.0	140.9	134.8	196.9	207.3	236.3	248.8	301
302	268.3	138.2	142.5	141.4	135.3	197.6	208.0	237.1	249.6	302
303	269.1	138.7	143.0	141.9	135.8	198.3	208.7	237.9	250.4	303
304	270.0	139.2	143.5	142.4	136.3	198.9	209.4	238.7	251.3	304
305	270.9	139.7	144.0	142.9	136.8	199.6	210.1	239.5	252.1	305
306	271.8	140.2	144.5	143.4	137.3	200.3	210.8	240.3	252.9	306
307	272.7	140.7	145.0	144.0	137.8	201.0	211.5	241.1	253.8	307
308	273.6	141.2	145.5	144.5	138.3	201.6	212.2	241.9	254.6	308
309	274.5	141.7	146.1	145.0	138.8	202.3	212.9	242.7	255.4	309
310	275.4	142.2	146.6	145.5	139.4	203.0	213.7	243.5	256.3	310
311	276.3	142.7	147.1	146.0	139.9	203.6	214.4	244.2	257.1	311
312	277.1	143.2	147.6	146.5	140.4	204.3	215.1	245.0	257.9	312
313	278.0	143.7	148.1	147.0	140.9	205.0	215.8	245.8	258.8	313
314	278.9	144.2	148.6	147.6	141.4	205.7	216.5	246.6	259.6	314
315	279.8	144.7	149.1	148.1	141.9	206.3	217.2	247.4	260.4	315
316	280.7	145.2	149.6	148.6	142.4	207.0	217.9	248.2	261.2	316
317	281.6	145.7	150.1	149.1	143.0	207.7	218.6	249.0	262.1	317
318	282.5	146.2	150.7	149.6	143.5	208.4	219.3	249.8	262.9	318
319	283.4	146.7	151.2	150.1	144.0	209.0	220.0	250.6	263.7	319
320	284.2	147.2	151.7	150.7	144.5	209.7	220.7	251.3	264.6	320
321	285.1	147.7	152.2	151.2	145.0	210.4	221.4	252.1	265.4	321
322	286.0	148.2	152.7	151.7	145.5	211.0	222.2	252.9	266.2	322
323	286.9	148.7	153.2	152.2	146.0	211.7	222.9	253.7	267.1	323
324	287.8	149.2	153.7	152.7	146.6	212.4	223.6	254.5	267.9	324
325	288.7	149.7	154.3	153.2	147.1	213.1	224.3	255.3	268.7	325
326	289.6	150.2	154.8	153.8	147.6	213.7	225.0	256.1	269.6	326
327	290.5	150.7	155.3	154.3	148.1	214.4	225.7	256.9	270.4	327
328	291.4	151.2	155.8	154.8	148.6	215.1	226.4	257.7	271.2	328
329	292.2	151.7	156.3	155.3	149.1	215.8	227.1	258.5	272.1	329
330	293.1	152.2	156.8	155.8	149.7	216.4	227.8	259.3	272.9	330
331	294.0	152.7	157.3	156.4	150.2	217.1	228.5	260.0	273.7	331
332	294.9	153.2	157.9	156.9	150.7	217.8	229.2	260.8	274.6	332
333	295.8	153.7	158.4	157.4	151.2	218.4	230.0	261.6	275.4	333
334	296.7	154.2	158.9	157.9	151.7	219.1	230.7	262.4	276.2	334
335	297.6	154.7	159.4	158.4	152.3	219.8	231.4	263.2	277.0	335
336	298.5	155.2	159.9	159.0	152.8	220.5	232.1	264.0	277.9	336
337	299.3	155.8	160.5	159.5	153.3	221.1	232.8	264.8	278.7	337
338	300.2	156.3	161.0	160.0	153.8	221.8	233.5	265.6	279.5	338
339	301.1	156.8	161.5	160.5	154.3	222.5	234.2	266.4	280.4	339

TABLE 1.—MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu ₂ O)	COPPER (Cu)	DEXTROSE (d-glucose)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu ₂ O)
				0.4 gram sugar	2 grams sugar	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ ·H ₂ O	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ ·H ₂ O	
340	302.0	157.3	162.0	161.0	154.8	222.2	224.9	267.1	281.2	340
341	302.9	157.8	162.5	161.6	155.4	222.8	225.6	267.9	282.0	341
342	303.8	158.3	163.1	162.1	155.9	223.5	226.3	268.7	282.9	342
343	304.7	158.8	163.6	162.6	156.4	224.2	227.0	269.5	283.7	343
344	305.6	159.3	164.1	163.1	156.9	224.9	227.8	270.3	284.5	344
345	306.5	159.8	164.6	163.7	157.5	225.5	228.5	271.1	285.4	345
346	307.3	160.3	165.1	164.2	158.0	227.2	229.2	271.9	286.2	346
347	308.2	160.8	165.7	164.7	158.5	227.9	229.9	272.7	287.0	347
348	309.1	161.4	166.2	165.2	159.0	228.5	230.6	273.5	287.9	348
349	310.0	161.9	166.7	165.7	159.5	229.2	231.3	274.3	288.7	349
350	310.9	162.4	167.2	166.3	160.1	229.9	232.0	275.0	289.5	350
351	311.8	162.9	167.7	166.8	160.6	230.6	232.7	275.8	290.4	351
352	312.7	163.4	168.3	167.3	161.1	231.3	233.4	276.6	291.2	352
353	313.6	163.9	168.8	167.8	161.6	231.9	234.1	277.4	292.0	353
354	314.4	164.4	169.3	168.4	162.2	232.6	234.8	278.2	292.8	354
355	315.3	164.9	169.8	168.9	162.7	233.3	235.6	279.0	293.7	355
356	316.2	165.4	170.4	169.4	163.2	233.9	236.3	279.8	294.5	356
357	317.1	166.0	170.9	170.0	163.7	234.6	237.0	280.6	295.3	357
358	318.0	166.5	171.4	170.5	164.3	235.3	237.7	281.4	296.2	358
359	318.9	167.0	171.9	171.0	164.8	236.0	238.4	282.2	297.0	359
360	319.8	167.5	172.5	171.5	165.3	236.7	239.1	282.9	297.8	360
361	320.7	168.0	173.0	172.1	165.8	237.3	239.8	283.7	298.7	361
362	321.6	168.5	173.5	172.6	166.4	238.0	240.5	284.5	299.5	362
363	322.4	169.0	174.0	173.1	166.9	238.7	241.2	285.3	300.3	363
364	323.3	169.6	174.6	173.7	167.4	239.4	242.0	286.1	301.2	364
365	324.2	170.1	175.1	174.2	167.9	240.0	242.7	286.9	302.0	365
366	325.1	170.6	175.6	174.7	168.5	240.7	243.4	287.7	302.8	366
367	326.0	171.1	176.1	175.2	169.0	241.4	244.1	288.5	303.6	367
368	326.9	171.6	176.7	175.8	169.5	242.1	244.8	289.3	304.5	368
369	327.8	172.1	177.2	176.3	170.0	242.7	245.5	290.0	305.3	369
370	328.7	172.7	177.7	176.8	170.6	243.4	246.2	290.8	306.1	370
371	329.5	173.2	178.3	177.4	171.1	244.1	246.9	291.6	307.0	371
372	330.4	173.7	178.8	177.9	171.6	244.8	247.7	292.4	307.8	372
373	331.3	174.2	179.3	178.4	172.2	245.4	248.4	293.2	308.6	373
374	332.2	174.7	179.8	179.0	172.7	246.1	249.1	294.0	309.5	374
375	333.1	175.3	180.4	179.5	173.2	246.8	249.8	294.8	310.3	375
376	334.0	175.8	180.9	180.0	173.7	247.5	250.5	295.6	311.1	376
377	334.9	176.3	181.4	180.6	174.3	248.1	251.2	296.4	312.0	377
378	335.8	176.8	182.0	181.1	174.8	248.8	251.9	297.2	312.8	378
379	336.7	177.3	182.5	181.6	175.3	249.5	252.6	297.9	313.6	379
380	337.5	177.9	183.0	182.1	175.9	250.2	253.4	298.7	314.5	380
381	338.4	178.4	183.6	182.7	176.4	250.8	254.1	299.5	315.3	381
382	339.3	178.9	184.1	183.2	176.9	251.5	254.8	300.3	316.1	382
383	340.2	179.4	184.6	183.8	177.5	252.2	255.5	301.1	316.9	383
384	341.1	180.0	185.2	184.3	178.0	252.9	256.2	301.9	317.8	384
385	342.0	180.5	185.7	184.8	178.5	253.6	256.9	302.7	318.6	385
386	342.9	181.0	186.2	185.4	179.1	254.2	257.6	303.5	319.4	386
387	343.8	181.5	186.8	185.9	179.6	254.9	258.3	304.3	320.3	387
388	344.6	182.0	187.3	186.4	180.1	255.6	259.0	305.0	321.1	388
389	345.5	182.6	187.8	187.0	180.6	256.3	259.8	305.8	321.9	389
390	346.4	183.1	188.4	187.5	181.2	256.9	260.5	306.6	322.8	390
391	347.3	183.6	188.9	188.0	181.7	257.6	261.2	307.4	323.6	391
392	348.2	184.1	189.4	188.6	182.3	258.3	261.9	308.2	324.4	392
393	349.1	184.7	190.0	189.1	182.8	259.0	262.6	309.0	325.2	393
394	350.0	185.2	190.5	189.7	183.3	259.6	263.3	309.8	326.1	394

TABLE 1.—MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPREOUS OXID (Cu ₂ O)	COPPER (Cu)	DEXTRINE (d-glucose)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPREOUS OXID (Cu ₂ O)
				0.4 gram sugar	2 grams sugar	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	
395	350.9	185.7	191.0	190.2	183.9	260.3	274.0	310.6	326.9	395
396	351.8	186.2	191.6	190.7	184.4	261.0	274.7	311.4	327.7	396
397	352.6	186.8	192.1	191.3	184.9	261.7	275.5	312.1	328.6	397
398	353.5	187.3	192.7	191.8	185.5	262.3	276.2	312.9	329.4	398
399	354.4	187.8	193.2	192.3	186.0	263.0	276.9	313.7	330.2	399
400	355.3	188.4	193.7	192.9	186.5	263.7	277.6	314.5	331.1	400
401	356.2	188.9	194.3	193.4	187.1	264.4	278.3	315.3	331.9	401
402	357.1	189.4	194.8	194.0	187.6	265.0	279.0	316.1	332.7	402
403	358.0	189.9	195.4	194.5	188.1	265.7	279.7	316.9	333.6	403
404	358.9	190.5	195.9	195.0	188.7	266.4	280.4	317.7	334.4	404
405	359.7	191.0	196.4	195.6	189.2	267.1	281.1	318.5	335.2	405
406	360.6	191.5	197.0	196.1	189.8	267.8	281.9	319.2	336.0	406
407	361.5	192.1	197.5	196.7	190.3	268.4	282.6	320.0	336.9	407
408	362.4	192.6	198.1	197.2	190.8	269.1	283.3	320.8	337.7	408
409	363.3	193.1	198.6	197.7	191.4	269.8	284.0	321.6	338.6	409
410	364.2	193.7	199.1	198.3	191.9	270.5	284.7	322.4	339.4	410
411	365.1	194.2	199.7	198.8	192.5	271.2	285.4	323.2	340.2	411
412	366.0	194.7	200.2	199.4	193.0	271.8	286.2	324.0	341.0	412
413	366.9	195.2	200.8	199.9	193.5	272.5	286.9	324.8	341.9	413
414	367.7	195.8	201.3	200.5	194.1	273.2	287.6	325.6	342.7	414
415	368.6	196.3	201.8	201.0	194.6	273.9	288.3	326.3	343.5	415
416	369.5	196.8	202.4	201.6	195.2	274.6	289.0	327.1	344.4	416
417	370.4	197.4	202.9	202.1	195.7	275.2	289.7	327.9	345.2	417
418	371.3	197.9	203.5	202.6	196.2	275.9	290.4	328.7	346.0	418
419	372.2	198.4	204.0	203.2	196.8	276.6	291.2	329.5	346.8	419
420	373.1	199.0	204.6	203.7	197.3	277.3	291.9	330.3	347.7	420
421	374.0	199.5	205.1	204.3	197.9	277.9	292.6	331.1	348.5	421
422	374.8	200.1	205.7	204.8	198.4	278.6	293.3	331.9	349.3	422
423	375.7	200.6	206.2	205.4	198.9	279.3	294.0	332.7	350.2	423
424	376.6	201.1	206.7	205.9	199.5	280.0	294.7	333.4	351.0	424
425	377.5	201.7	207.3	206.5	200.0	280.7	295.4	334.2	351.8	425
426	378.4	202.2	207.8	207.0	200.6	281.3	296.2	335.0	352.7	426
427	379.3	202.8	208.4	207.6	201.1	282.0	296.9	335.8	353.5	427
428	380.2	203.3	208.9	208.1	201.7	282.7	297.6	336.6	354.3	428
429	381.1	203.8	209.5	208.7	202.3	283.4	298.3	337.4	355.1	429
430	382.0	204.4	210.0	209.2	202.7	284.1	299.0	338.2	356.0	430
431	382.8	204.9	210.6	209.8	203.3	284.7	299.7	339.0	356.8	431
432	383.7	205.5	211.1	210.3	203.8	285.4	300.5	339.7	357.6	432
433	384.6	206.0	211.7	210.9	204.4	286.1	301.2	340.5	358.5	433
434	385.5	206.5	212.2	211.4	204.9	286.8	301.9	341.3	359.3	434
435	386.4	207.1	212.8	212.0	205.5	287.5	302.6	342.1	360.1	435
436	387.3	207.6	213.3	212.5	206.0	288.1	303.3	342.9	361.0	436
437	388.2	208.2	213.9	213.1	206.6	288.8	304.0	343.7	361.8	437
438	389.1	208.7	214.4	213.6	207.1	289.5	304.7	344.5	362.6	438
439	390.0	209.2	215.0	214.2	207.7	290.2	305.5	345.3	363.4	439
440	390.8	209.8	215.5	214.7	208.2	290.9	306.2	346.1	364.3	440
441	391.7	210.3	216.1	215.3	208.8	291.5	306.9	346.8	365.1	441
442	392.6	210.9	216.6	215.8	209.3	292.2	307.6	347.6	365.9	442
443	393.5	211.4	217.2	216.4	209.9	292.9	308.3	348.4	366.8	443
444	394.4	212.0	217.8	216.9	210.4	293.6	309.0	349.2	367.6	444
445	395.3	212.5	218.3	217.5	211.0	294.2	309.7	350.0	368.4	445
446	396.2	213.1	218.9	218.0	211.5	294.9	310.5	350.8	369.3	446
447	397.1	213.6	219.4	218.6	212.1	295.6	311.2	351.6	370.1	447
448	397.9	214.1	220.0	219.1	212.6	296.3	311.9	352.4	370.9	448
449	398.8	214.7	220.5	219.7	213.2	297.0	312.6	353.2	371.7	449

TABLE 1.—MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu ₂ O)	COPPER (Cu)	DEXTROSE (d-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu ₂ O)
				0.4 gram sugar	2 grams sugar	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	
450	399.7	215.2	221.1	220.2	213.7	297.6	313.3	353.9	372.6	450
451	400.6	215.8	221.6	220.8	214.3	298.3	314.0	354.7	373.4	451
452	401.5	216.3	222.2	221.4	214.8	299.0	314.7	355.5	374.2	452
453	402.4	216.9	222.8	221.9	215.4	299.7	315.5	356.3	375.1	453
454	403.3	217.4	223.3	222.5	215.9	300.4	316.2	357.1	375.9	454
455	404.2	218.0	223.9	223.0	216.5	301.1	316.9	357.9	376.7	455
456	405.1	218.5	224.4	223.6	217.0	301.7	317.6	358.7	377.6	456
457	405.9	219.1	225.0	224.1	217.6	302.4	318.3	359.5	378.4	457
458	406.8	219.6	225.5	224.7	218.1	303.1	319.0	360.3	379.2	458
459	407.7	220.2	226.1	225.3	218.7	303.8	319.8	361.0	380.0	459
460	408.6	220.7	226.7	225.8	219.2	304.5	320.5	361.8	380.9	460
461	409.5	221.3	227.2	226.4	219.8	305.1	321.2	362.6	381.7	461
462	410.4	221.8	227.8	226.9	220.3	305.8	321.9	363.4	382.5	462
463	411.3	222.4	228.3	227.5	220.9	306.5	322.6	364.2	383.4	463
464	412.2	222.9	228.9	228.1	221.4	307.2	323.4	365.0	384.2	464
465	413.0	223.5	229.5	228.6	222.0	307.9	324.1	365.8	385.0	465
466	413.9	224.0	230.0	229.2	222.5	308.6	324.8	366.6	385.9	466
467	414.8	224.6	230.6	229.7	223.1	309.2	325.5	367.3	386.7	467
468	415.7	225.1	231.2	230.3	223.7	309.9	326.2	368.1	387.5	468
469	416.6	225.7	231.7	230.9	224.2	310.6	326.9	368.9	388.3	469
470	417.5	226.2	232.3	231.4	224.8	311.3	327.7	369.7	389.2	470
471	418.4	226.8	232.8	232.0	225.3	312.0	328.4	370.5	390.0	471
472	419.3	227.4	233.4	232.5	225.9	312.6	329.1	371.3	390.8	472
473	420.2	227.9	234.0	233.1	226.4	313.3	329.8	372.1	391.7	473
474	421.0	228.5	234.5	233.7	227.0	314.0	330.5	372.9	392.5	474
475	421.9	229.0	235.1	234.2	227.6	314.7	331.3	373.7	393.3	475
476	422.8	229.6	235.7	234.8	228.1	315.4	332.0	374.4	394.2	476
477	423.7	230.1	236.2	235.4	228.7	316.1	332.7	375.2	395.0	477
478	424.6	230.7	236.8	235.9	229.2	316.7	333.4	376.0	395.8	478
479	425.5	231.3	237.4	236.5	229.8	317.4	334.1	376.8	396.6	479
480	426.4	231.8	237.9	237.1	230.3	318.1	334.8	377.6	397.5	480
481	427.3	232.4	238.5	237.6	230.9	318.8	335.6	378.4	398.3	481
482	428.1	232.9	239.1	238.2	231.5	319.5	336.3	379.2	399.1	482
483	429.0	233.5	239.6	238.8	232.0	320.1	337.0	380.0	400.0	483
484	429.9	234.1	240.2	239.3	232.6	320.8	337.7	380.7	400.8	484
485	430.8	234.6	240.8	239.9	233.2	321.5	338.4	381.5	401.6	485
486	431.7	235.2	241.4	240.5	233.7	322.2	339.1	382.3	402.4	486
487	432.6	235.7	241.9	241.0	234.3	322.9	339.9	383.1	403.3	487
488	433.5	236.3	242.5	241.6	234.8	323.6	340.6	383.9	404.1	488
489	434.4	236.9	243.1	242.2	235.4	324.2	341.3	384.7	404.9	489
490	435.3	237.4	243.6	242.7	236.0	324.9	342.0	385.5	405.8	490

II. A. H. Low Volumetric Method, Modified.⁴—Tentative.

REAGENT.

Standard thiosulphate solution.—Prepare a solution of sodium thiosulphate containing 19 grams of pure crystals in 1 liter. Weigh accurately about 0.2 gram of pure copper foil and place in a flask of 250 cc. capacity. Dissolve by warming with 5 cc. of a mixture of equal volumes of strong nitric acid and water. Dilute to 50 cc., boil to expel the red fumes, add 5 cc. of strong bromin water, and boil

until the bromin is completely driven off. Remove from the heat and add a slight excess of strong ammonium hydroxid (about 7 cc. is required). Again boil until the excess of ammonia is expelled, as shown by a change of color of the liquid, and a partial precipitation. Then add a slight excess of strong acetic acid (3 or 4 cc. of 80% acid) and boil for a minute. Cool to room temperature and add 10 cc. of 30% potassium iodid solution. Titrate at once with the thiosulphate solution until the brown tinge has become weak, then add sufficient starch indicator (VII, 3 (a)) to produce a marked blue coloration. Continue the titration cautiously until the color due to free iodin has entirely vanished. The blue color changes toward the end to a faint lilac. If at this point the thiosulphate be added drop by drop and a little time allowed for complete reaction after each addition, there is no difficulty in determining the end point within a single drop. One cc. of the thiosulphate solution will be found to correspond to about 0.005 gram of copper.

29

DETERMINATION.

After washing the precipitated cuprous oxid, cover the Gooch with a watch glass and dissolve the oxid by means of 5 cc. of warm nitric acid (1 to 1) poured under the watch glass with a pipette. Catch the filtrate in a 250 cc. flask, wash the watch glass and Gooch free of copper, using about 50 cc. of water. Boil to expel red fumes, add 5 cc. of bromin water, boil off the bromin, and proceed exactly as in 28.

30

III. Volumetric Permanganate Method.—Tentative.

Filter and wash the cuprous oxid as directed under 25. Transfer the asbestos film to the beaker, add about 30 cc. of hot water, and beat the precipitate and asbestos thoroughly. Rinse the crucible with 50 cc. of a hot saturated solution of ferri sulphate in 20% sulphuric acid, receiving the rinsings in the beaker containing the precipitate. After the cuprous oxid is dissolved, wash the solution into a large Erlenmeyer flask and immediately titrate with a standard solution of potassium permanganate, 1 cc. of which should be equivalent to 0.010 gram of copper. Standardize this solution by making 6 or more determinations with the same sugar solution, titrating one half of the precipitates obtained, and determining the copper in the others by electrolysis. The average weight of copper obtained by electrolysis, divided by the average number of cc. of permanganate solution required for the titrations, gives the weight of copper equivalent to 1 cc. of the standard permanganate solution. A solution standardized with iron or oxalic acid will give too low a result.

31

IV. Electrolytic Deposition from Sulphuric Acid Solution.—Tentative.

Filter the cuprous oxid in a Gooch, wash the beaker and the precipitate thoroughly with hot water without transferring the precipitate to the filter. Wash the asbestos film and the adhering cuprous oxid into the beaker by means of hot dilute nitric acid. After the copper is all in solution, refilter through a thin film of asbestos in a Gooch and wash thoroughly with hot water. Add 10 cc. of sulphuric acid (1 to 4), and evaporate the filtrate on the steam bath until the copper salt has largely crystallized. Heat carefully on a hot plate or over asbestos until the evolution of white fumes shows that the excess of nitric acid is removed. Add 8-10 drops of nitric acid (sp. gr. 1.42) and rinse into a 100-125 cc. platinum dish. Deposit the copper by

electrolysis. Wash thoroughly with water, then break the current, wash with alcohol and ether successively, dry at about 50°C., and weigh. If preferred, the electrolysis can be conducted in a beaker, the copper being deposited upon a weighed platinum electrode.

32 *V. Electrolytic Deposition from Sulphuric and Nitric Acid Solution.—Tentative.*

Filter and wash as directed under 31. Transfer the asbestos film from the crucible to the beaker by means of a glass rod and rinse the crucible with about 30 cc. of a boiling mixture of dilute sulphuric and nitric acids, containing 65 cc. of sulphuric acid (sp. gr. 1.84) and 50 cc. of nitric acid (sp. gr. 1.42) per liter. Heat and agitate until solution is complete; filter and electrolyze as under 31.

33 *VI. Electrolytic Deposition from Nitric Acid Solution.—Tentative.*

Filter and wash as directed under 31. Transfer the asbestos film and adhering oxid to the beaker. Dissolve the oxid still remaining in the crucible by means of 2 cc. of nitric acid (sp. gr. 1.42), adding it with a pipette and receiving the solution in the beaker containing the asbestos film. Rinse the crucible with a jet of water, allowing the rinsings to flow into the beaker. Heat the contents of the beaker until the copper is all in solution, filter, dilute the filtrate to a volume of 100 cc. or more, and electrolyze. When a nitrate solution is electrolyzed, the first washing of the deposit should be made with water acidulated with sulphuric acid, in order to remove all the nitric acid before the current is interrupted.

34 *VII. Reduction in Hydrogen.—Tentative.*

Deposit an asbestos film on a perforated platinum disc or cone contained in a hard glass filtering tube, wash free from loose fibers, dry and weigh. Through this tube, previously moistened, filter the cuprous oxid immediately, using suction. Transfer the cuprous oxid to the tube through a removable funnel, and wash thoroughly with hot water, alcohol and ether successively. After drying, connect the tube with a supply of dry hydrogen, heat gently until the cuprous oxid is completely reduced to metallic copper, cool in the current of hydrogen, and weigh. If preferred, a Gooch crucible may be used for the filtration.

Herzfeld Gravimetric Method.—Tentative.

Method I.

(For materials containing 1.5% or less of invert sugar and 98.5% or more of sucrose.)

35

REAGENTS.

The reagents and solutions used are described under 24.

36

DETERMINATION.

Prepare the solution of the material to be examined so as to contain 20 grams in 100 cc., free from suspended impurities by filtration and from soluble impurities by neutral lead acetate, removing the excess of lead by means of sodium carbonate. Place 50 cc. of the reagent and 50 cc. of the sugar solution in a 250 cc. beaker. Heat this mixture at such a rate that approximately 4 minutes are required to bring it to the boiling point, and boil for exactly 2 minutes. Add 100 cc. of cold, recently boiled, water. Filter immediately through asbestos, and determine the copper by one of the methods under 26, 29-34, respectively. Obtain the corresponding percentage of invert sugar from 37.

37

TABLE 2.—HERZFELD'S TABLE.⁵

For the determination of invert sugar in materials containing 1.5%, or less, of invert sugar and 98.5%, or more, of sucrose.

COPPER REDUCED BY 10 GRAMS OF MATERIAL	INVERT SUGAR	COPPER REDUCED BY 10 GRAMS OF MATERIAL	INVERT SUGAR	COPPER REDUCED BY 10 GRAMS OF MATERIAL	INVERT SUGAR
mg.	per cent	mg.	per cent	mg.	per cent
50	0.05	140	0.51	230	1.02
55	0.07	145	0.53	235	1.05
60	0.09	150	0.56	240	1.07
65	0.11	155	0.59	245	1.10
70	0.14	160	0.62	250	1.13
75	0.16	165	0.65	255	1.16
80	0.19	170	0.68	260	1.18
85	0.21	175	0.71	265	1.21
90	0.24	180	0.74	270	1.24
95	0.27	185	0.76	275	1.27
100	0.30	190	0.79	280	1.30
105	0.32	195	0.82	285	1.33
110	0.35	200	0.85	290	1.36
115	0.38	205	0.88	295	1.38
120	0.40	210	0.90	300	1.41
125	0.43	215	0.93	305	1.44
130	0.45	220	0.96	310	1.47
135	0.48	225	0.99	315	1.50

Method II.

(For materials containing 1.5% or more of invert sugar and 98.5% or less of sucrose.)

38

REAGENTS.

Same as described under 24.

39

DETERMINATION.

Prepare a solution of the material to be examined in such a manner that it contains 20 grams in 100 cc. after clarification and removal of the excess of lead. Prepare a series of solutions in large test tubes by adding 1, 2, 3, 4, and 5 cc. of this solution to each tube successively. Add 5 cc. of the reagent to each, heat to boiling, boil 2 minutes, and filter. Note the volume of sugar solution which gives the filtrate lightest in tint, but still distinctly blue. Place 20 times this volume of the sugar solution in a 100 cc. flask, dilute to the mark, and mix well. Use 50 cc. of the solution for the determination, which is conducted as described under 36. For the calculation of the result use the following formulas and table of factors of Meissl and Hiller:

Let Cu = the weight of copper obtained;

P = the polarization of the sample;

W = the weight of the sample in the 50 cc. of the solution used for the determination;

F = the factor obtained from the table for the conversion of copper to invert sugar;

Then $\frac{Cu}{2} = Z$, approximate weight of invert sugar;

$Z \times \frac{100}{W} = Y$, approximate per cent of invert sugar;

$$\frac{100 P}{P + Y} = R, \text{ approximate per cent of sucrose in mixture of sugars;}$$

$$100 - R = I, \text{ approximate per cent of invert sugar;}$$

$$\frac{\text{CuF}}{W} = \text{per cent of invert sugar.}$$

The factor F for calculating copper to invert sugar is then found from 40.

40

TABLE 3.

Meissl and Hiller's⁶ factors for determinations in materials in which, of the total sugars present, 1.5%, or more, is invert sugar, and 98.5%, or less, is sucrose.

RATIO OF SUCROSE TO INVERT SUGAR = R:I.	APPROXIMATE ABSOLUTE WEIGHT OF INVERT SUGAR (Z)						
	200 milligrams	175 milligrams	150 milligrams	125 milligrams	100 milligrams	75 milligrams	50 milligrams
	per cent	per cent	per cent	per cent	per cent	per cent	per cent
0:100	56.4	55.4	54.5	53.8	53.2	53.0	53.0
10:90	56.3	55.3	54.4	53.8	53.2	52.9	52.9
20:80	56.2	55.2	54.3	53.7	53.2	52.7	52.7
30:70	56.1	55.1	54.2	53.7	53.2	52.6	52.6
40:60	55.9	55.0	54.1	53.6	53.1	52.5	52.4
50:50	55.7	54.9	54.0	53.5	53.1	52.3	52.2
60:40	55.6	54.7	53.8	53.2	52.8	52.1	51.9
70:30	55.5	54.5	53.5	52.9	52.5	51.9	51.6
80:20	55.4	54.3	53.3	52.7	52.2	51.7	51.3
90:10	54.6	53.6	53.1	52.6	52.1	51.6	51.2
91:9	54.1	53.6	52.6	52.1	51.6	51.2	50.7
92:8	53.6	53.1	52.1	51.6	51.2	50.7	50.3
93:7	53.6	53.1	52.1	51.2	50.7	50.3	49.8
94:6	53.1	52.6	51.6	50.7	50.3	49.8	48.9
95:5	52.6	52.1	51.2	50.3	49.4	48.9	48.5
96:4	52.1	51.2	50.7	49.8	48.9	47.7	46.9
97:3	50.7	50.3	49.8	48.9	47.7	46.2	45.1
98:2	49.9	48.9	48.5	47.3	45.8	43.3	40.0
99:1	47.7	47.3	46.5	45.1	43.3	41.2	38.1

Example: The polarization of a sugar is 86.4, and 50 cc. of solution containing 3.256 grams of sample gave 0.290 gram of copper.

$$\frac{\text{Cu}}{2} = \frac{0.290}{2} = 0.145 = Z$$

$$\frac{Z \times 100}{W} = 0.145 \times \frac{100}{3.256} = 4.45 = Y$$

$$100 - R = 100 - 95.1 = I = 4.9$$

$$R:I = 95.1:4.9$$

By consulting the table it will be seen that the vertical column headed 150 is nearest to Z, 145, and the horizontal column headed 95:5 is nearest to the ratio of R to I, 95.1:4.9. Where these columns meet, we find the factor 51.2 which enters into the final calculation:

$$\frac{\text{CuF}}{W} = \frac{0.290 \times 51.2}{3.256} = 4.56 \text{ per cent of invert sugar.}$$

In case there is no sucrose present, the following table may be used instead of the factors given in 40.

41

TABLE 4.—MEISSL'S TABLE.⁷*For the determination of invert sugar alone.*

[According to Wein.]

[Expressed in milligrams.]

COPPER	INVERT SUGAR	COPPER	INVERT SUGAR	COPPER	INVERT SUGAR	COPPER	INVERT SUGAR
90	46.9	135	70.8	180	95.2	225	120.4
91	47.4	136	71.3	181	95.7	226	120.9
92	47.9	137	71.9	182	96.2	227	121.5
93	48.4	138	72.4	183	96.8	228	122.1
94	48.9	139	72.9	184	97.3	229	122.6
95	49.5	140	73.5	185	97.8	230	123.2
96	50.0	141	74.0	186	98.4	231	123.8
97	50.5	142	74.5	187	99.0	232	124.3
98	51.1	143	75.1	188	99.5	233	124.9
99	51.6	144	75.6	189	100.1	234	125.5
100	52.1	145	76.1	190	100.6	235	126.0
101	52.7	146	76.7	191	101.2	236	126.6
102	53.2	147	77.2	192	101.7	237	127.2
103	53.7	148	77.8	193	102.3	238	127.8
104	54.3	149	78.3	194	102.9	239	128.3
105	54.8	150	78.9	195	103.4	240	128.9
106	55.3	151	79.4	196	104.0	241	129.5
107	55.9	152	80.0	197	104.6	242	130.0
108	56.4	153	80.5	198	105.1	243	130.6
109	56.9	154	81.0	199	105.7	244	131.2
110	57.5	155	81.6	200	106.3	245	131.8
111	58.0	156	82.1	201	106.8	246	132.3
112	58.5	157	82.7	202	107.4	247	132.9
113	59.1	158	83.2	203	107.9	248	133.5
114	59.6	159	83.8	204	108.5	249	134.1
115	60.1	160	84.3	205	109.1	250	134.6
116	60.7	161	84.8	206	109.6	251	135.2
117	61.2	162	85.4	207	110.2	252	135.8
118	61.7	163	85.9	208	110.8	253	136.3
119	62.3	164	86.5	209	111.3	254	136.9
120	62.8	165	87.0	210	111.9	255	137.5
121	63.3	166	87.6	211	112.5	256	138.1
122	63.9	167	88.1	212	113.0	257	138.6
123	64.4	168	88.6	213	113.6	258	139.2
124	64.9	169	89.2	214	114.2	259	139.8
125	65.5	170	89.7	215	114.7	260	140.4
126	66.0	171	90.3	216	115.3	261	140.9
127	66.5	172	90.8	217	115.8	262	141.5
128	67.1	173	91.4	218	116.4	263	142.1
129	67.6	174	91.9	219	117.0	264	142.7
130	68.1	175	92.4	220	117.5	265	143.2
131	68.7	176	93.0	221	118.1	266	143.8
132	69.2	177	93.5	222	118.7	267	144.4
133	69.7	178	94.1	223	119.2	268	144.9
134	70.3	179	94.6	224	119.8	269	145.5

TABLE 4.—MEISSL'S TABLE.—Continued.

(Expressed in milligrams.)

COPPER	INVERT SUGAR	COPPER	INVERT SUGAR	COPPER	INVERT SUGAR	COPPER	INVERT SUGAR
270	146.1	310	169.7	350	193.8	390	218.7
271	146.7	311	170.3	351	194.4	391	219.3
272	147.2	312	170.9	352	195.0	392	219.9
273	147.8	313	171.5	353	195.6	393	220.5
274	148.4	314	172.1	354	196.2	394	221.2
275	149.0	315	172.7	355	196.8	395	221.8
276	149.5	316	173.3	356	197.4	396	222.4
277	150.1	317	173.9	357	198.0	397	223.1
278	150.7	318	174.5	358	198.6	398	223.7
279	151.3	319	175.1	359	199.2	399	224.3
280	151.9	320	175.6	360	199.8	400	224.9
281	152.5	321	176.2	361	200.4	401	225.7
282	153.1	322	176.8	362	201.1	402	226.4
283	153.7	323	177.4	363	201.7	403	227.1
284	154.3	324	178.0	364	202.3	404	227.8
285	154.9	325	178.6	365	203.0	405	228.6
286	155.5	326	179.2	366	203.6	406	229.3
287	156.1	327	179.8	367	204.2	407	230.0
288	156.7	328	180.4	368	204.8	408	230.7
289	157.2	329	181.0	369	205.5	409	231.4
290	157.8	330	181.6	370	206.1	410	232.1
291	158.4	331	182.2	371	206.7	411	232.8
292	159.0	332	182.8	372	207.3	412	233.5
293	159.6	333	183.5	373	208.0	413	234.3
294	160.2	334	184.1	374	208.6	414	235.0
295	160.8	335	184.7	375	209.2	415	235.7
296	161.4	336	185.4	376	209.9	416	236.4
297	162.0	337	186.0	377	210.5	417	237.1
298	162.6	338	186.6	378	211.1	418	237.8
299	163.2	339	187.2	379	211.7	419	238.5
300	163.8	340	187.8	380	212.4	420	239.2
301	164.4	341	188.4	381	213.0	421	239.9
302	165.0	342	189.0	382	213.6	422	240.6
303	165.6	343	189.6	383	214.3	423	241.3
304	166.2	344	190.2	384	214.9	424	242.0
305	166.8	345	190.8	385	215.5	425	242.7
306	167.3	346	191.4	386	216.1	426	243.4
307	167.9	347	192.0	387	216.8	427	244.1
308	168.5	348	192.6	388	217.4	428	244.9
309	169.1	349	193.2	389	218.0	429	245.6
						430	246.3

MALTOSE.

General Gravimetric Method.—Tentative.

Proceed as directed under 25 and obtain, from 27, the weight of maltose equivalent to the weight of copper reduced.

Wein Method.—Tentative.

43

REAGENTS.

The reagents and solutions used are described under 24.

44

DETERMINATION.

Place 50 cc. of the reagent in a beaker and heat to the boiling point. When boiling briskly, add 25 cc. of the maltose solution containing not more than 0.250 gram of maltose and boil for 4 minutes. Filter immediately through asbestos and determine, by one of the methods given under 26, 29-34 respectively, the amount of copper reduced.

Obtain, from 45, the weight of maltose equivalent to the weight of copper found.

45

TABLE 5.

For the determination of maltose.

[According to Wein.*]

[Expressed in milligrams.]

COPPER	CU- PROUS OXID	MAL- TOSE	COPPER	CU- PROUS OXID	MAL- TOSE	COPPER	CU- PROUS OXID	MAL- TOSE	COPPER	CU- PROUS OXID	MAL- TOSE
31	34.9	26.1	71	79.9	61.0	111	125.0	96.4	151	170.0	132.3
32	36.0	27.0	72	81.1	61.8	112	126.1	97.3	152	171.1	133.2
33	37.2	27.9	73	82.2	62.7	113	127.2	98.1	153	172.3	134.1
34	38.3	28.7	74	83.3	63.6	114	128.3	99.0	154	173.4	135.0
35	39.4	29.6	75	84.4	64.5	115	129.6	99.9	155	174.5	135.9
36	40.5	30.5	76	85.6	65.4	116	130.6	100.8	156	175.6	136.8
37	41.7	31.3	77	86.7	66.2	117	131.7	101.7	157	176.8	137.7
38	42.8	32.2	78	87.8	67.1	118	132.8	102.6	158	177.9	138.6
39	43.9	33.1	79	88.9	68.0	119	134.0	103.6	159	179.0	139.5
40	45.0	33.9	80	90.1	68.9	120	135.1	104.4	160	180.1	140.4
41	46.2	34.8	81	91.2	69.7	121	136.2	105.3	161	181.3	141.3
42	47.3	35.7	82	92.3	70.6	122	137.4	106.2	162	182.4	142.2
43	48.4	36.6	83	93.4	71.5	123	138.5	107.1	163	183.5	143.1
44	49.5	37.4	84	94.6	72.4	124	139.6	108.0	164	184.6	144.0
45	50.7	38.3	85	95.7	73.2	125	140.7	108.9	165	185.8	144.9
46	51.8	39.1	86	96.8	74.1	126	141.9	109.8	166	186.9	145.8
47	52.9	40.0	87	97.9	75.0	127	143.0	110.7	167	188.0	146.7
48	54.0	40.9	88	99.1	75.9	128	144.1	111.6	168	189.1	147.6
49	55.2	41.8	89	100.2	76.8	129	145.2	112.5	169	190.3	148.5
50	56.3	42.6	90	101.3	77.7	130	146.4	113.4	170	191.4	149.4
51	57.4	43.5	91	102.4	78.6	131	147.5	114.3	171	192.5	150.3
52	58.5	44.4	92	103.6	79.5	132	148.6	115.2	172	193.6	151.2
53	59.7	45.2	93	104.7	80.3	133	149.7	116.1	173	194.8	152.0
54	60.8	46.1	94	105.8	81.2	134	150.9	117.0	174	195.9	152.9
55	61.9	47.0	95	107.0	82.1	135	152.0	117.9	175	197.0	153.8
56	63.0	47.8	96	108.1	83.0	136	153.1	118.8	176	198.1	154.7
57	64.2	48.7	97	109.2	83.9	137	154.2	119.7	177	199.3	155.6
58	65.3	49.6	98	110.3	84.8	138	155.4	120.6	178	200.4	156.5
59	66.4	50.4	99	111.5	85.7	139	156.5	121.5	179	201.5	157.4
60	67.6	51.3	100	112.6	86.6	140	157.6	122.4	180	202.6	158.3
61	68.7	52.2	101	113.7	87.5	141	158.7	123.3	181	203.8	159.2
62	69.8	53.1	102	114.8	88.4	142	159.9	124.2	182	204.9	160.1
63	70.9	53.9	103	116.0	89.2	143	161.0	125.1	183	206.0	160.9
64	72.1	54.8	104	117.1	90.1	144	162.1	126.0	184	207.1	161.8
65	73.2	55.7	105	118.2	91.0	145	163.2	126.9	185	208.3	162.7
66	74.3	56.6	106	119.3	91.9	146	164.4	127.8	186	209.4	163.6
67	75.4	57.4	107	120.5	92.8	147	165.5	128.7	187	210.5	164.5
68	76.6	58.3	108	121.6	93.7	148	166.6	129.6	188	211.7	165.4
69	77.7	59.2	109	122.7	94.6	149	167.7	130.5	189	212.8	166.3
70	78.8	60.1	110	123.8	95.5	150	168.9	131.4	190	213.9	167.2

TABLE 5.—Continued.
For the determination of maltose.
[Expressed in milligrams.]

COFFER	CU- PROUS OXID	MAL- TOSE	COFFER	CU- PROUS OXID	MAL- TOSE	COFFER	CU- PROUS OXID	MAL- TOSE	COFFER	CU- PROUS OXID	MAL- TOSE
191	215.0	168.1	221	243.7	194.8	251	282.6	221.7	281	316.4	248.7
192	216.2	169.0	222	249.9	195.7	252	283.7	222.6	282	317.5	249.6
193	217.3	169.8	223	251.0	196.6	253	284.8	223.5	283	318.6	250.4
194	218.4	170.7	224	252.4	197.5	254	286.0	224.4	284	319.7	251.3
195	219.5	171.6	225	253.3	198.4	255	287.1	225.3	285	320.9	252.3
196	220.7	172.5	226	254.4	199.3	256	288.2	226.2	286	322.0	253.1
197	221.8	173.4	227	255.6	200.2	257	289.3	227.1	287	323.1	254.0
198	222.9	174.3	228	256.7	201.1	258	290.5	228.0	288	324.2	254.9
199	224.0	175.2	229	257.8	202.0	259	291.6	228.9	289	325.4	255.8
200	225.2	176.1	230	258.9	202.9	260	292.7	229.8	290	326.5	256.6
201	226.3	177.0	231	260.1	203.8	261	293.8	230.7	291	327.4	257.5
202	227.4	177.9	232	261.2	204.7	262	295.0	231.6	292	328.7	258.4
203	228.5	178.7	233	262.3	205.6	263	296.1	232.5	293	329.9	259.3
204	229.7	179.6	234	263.4	206.5	264	297.2	233.4	294	331.0	260.3
205	230.8	180.5	235	264.6	207.4	265	298.3	234.3	295	332.1	261.1
206	231.9	181.4	236	265.7	208.3	266	299.5	235.2	296	333.2	262.0
207	233.0	182.3	237	266.8	209.1	267	300.6	236.1	297	334.4	262.8
208	234.2	183.2	238	268.0	210.0	268	301.7	237.0	298	335.5	263.7
209	235.3	184.1	239	269.1	210.9	269	302.8	237.9	299	336.6	264.6
210	236.4	185.0	240	270.2	211.8	270	304.0	238.8	300	337.8	265.5
211	237.6	185.9	241	271.3	212.7	271	305.1	239.7			
212	238.7	186.8	242	272.5	213.6	272	306.2	240.6			
213	239.8	187.7	243	273.6	214.5	273	307.3	241.5			
214	240.9	188.6	244	274.7	215.4	274	308.5	242.4			
215	242.1	189.5	245	275.8	216.3	275	309.6	243.3			
216	243.2	190.4	246	277.0	217.2	276	310.7	244.2			
217	244.3	191.2	247	278.1	218.1	277	311.9	245.1			
218	245.4	192.1	248	279.2	219.0	278	313.0	246.0			
219	246.6	193.0	249	280.3	219.9	279	314.1	246.9			
220	247.7	193.9	250	281.5	220.8	280	315.2	247.8			

LACTOSE.

General Gravimetric Method.—Tentative.

Proceed as directed under 25 and obtain, from 27, the weight of lactose equivalent to the weight of copper reduced.

Soxhlet-Wein Method.—Official.

REAGENTS.

The reagents and solutions used are described under 24.

DETERMINATION.

Place 50 cc. of the reagent in a beaker and heat to the boiling point. When boiling briskly, add 100 cc. of the lactose solution containing not more than 0.300 gram of lactose and boil for 6 minutes. Filter immediately through asbestos and determine by one of the methods given under 26, 29-34 inclusive, the amount of copper reduced. Obtain, from 49, the weight of lactose equivalent to the weight of copper found.

49

TABLE 6.

For the determination of lactose (Soxhlet-Wein⁹).

[Expressed in milligrams.]

COFFER	LACTOSE	COFFER	LACTOSE	COFFER	LACTOSE	COFFER	LACTOSE	COFFER	LACTOSE
100	71.6	160	116.4	220	161.9	280	208.3	340	255.7
101	72.4	161	117.1	221	162.7	281	209.1	341	256.5
102	73.1	162	117.9	222	163.4	282	209.9	342	257.4
103	73.8	163	118.6	223	164.2	283	210.7	343	258.2
104	74.6	164	119.4	224	164.9	284	211.5	344	259.0
105	75.3	165	120.2	225	165.7	285	212.3	345	259.8
106	76.1	166	120.9	226	166.4	286	213.1	346	260.6
107	76.8	167	121.7	227	167.2	287	213.9	347	261.4
108	77.6	168	122.4	228	167.9	288	214.7	348	262.3
109	78.3	169	123.2	229	168.6	289	215.5	349	263.1
110	79.0	170	123.9	230	169.4	290	216.3	350	263.9
111	79.8	171	124.7	231	170.1	291	217.1	351	264.7
112	80.5	172	125.5	232	170.9	292	217.9	352	265.5
113	81.3	173	126.2	233	171.6	293	218.7	353	266.3
114	82.0	174	127.0	234	172.4	294	219.5	354	267.2
115	82.7	175	127.8	235	173.1	295	220.3	355	268.0
116	83.5	176	128.5	236	173.9	296	221.1	356	268.8
117	84.2	177	129.3	237	174.6	297	221.9	357	269.6
118	85.0	178	130.1	238	175.4	298	222.7	358	270.4
119	85.7	179	130.8	239	176.2	299	223.5	359	271.2
120	86.4	180	131.6	240	176.9	300	224.4	360	272.1
121	87.2	181	132.4	241	177.7	301	225.2	361	272.9
122	87.9	182	133.1	242	178.5	302	225.9	362	273.7
123	88.7	183	133.9	243	179.3	303	226.7	363	274.5
124	89.4	184	134.7	244	180.1	304	227.5	364	275.3
125	90.1	185	135.4	245	180.8	305	228.3	365	276.2
126	90.9	186	136.2	246	181.6	306	229.1	366	277.1
127	91.6	187	137.0	247	182.4	307	229.8	367	277.9
128	92.4	188	137.7	248	183.2	308	230.6	368	278.8
129	93.1	189	138.5	249	184.0	309	231.4	369	279.6
130	93.8	190	139.3	250	184.8	310	232.2	370	280.5
131	94.6	191	140.0	251	185.5	311	232.9	371	281.4
132	95.3	192	140.8	252	186.3	312	243.7	372	282.2
133	96.1	193	141.6	253	187.1	313	234.5	373	283.1
134	96.9	194	142.3	254	187.9	314	235.3	374	283.9
135	97.6	195	143.1	255	188.7	315	236.1	375	284.8
136	98.3	196	143.9	256	189.4	316	236.8	376	285.7
137	99.1	197	144.6	257	190.2	317	237.6	377	286.5
138	99.8	198	145.4	258	191.0	318	238.4	378	287.4
139	100.5	199	146.2	259	191.8	319	239.2	379	288.2
140	101.3	200	146.9	260	192.5	320	240.0	380	289.1
141	102.0	201	147.7	261	193.3	321	240.7	381	289.9
142	102.8	202	148.5	262	194.1	322	241.5	382	290.8
143	103.5	203	149.2	263	194.9	323	242.3	383	291.7
144	104.3	204	150.0	264	195.7	324	243.1	384	292.5
145	105.1	205	150.7	265	196.4	325	243.9	385	293.4
146	105.8	206	151.5	266	197.2	326	244.6	386	294.2
147	106.6	207	152.2	267	198.0	327	245.4	387	295.1
148	107.3	208	153.0	268	198.8	328	246.2	388	296.0
149	108.1	209	153.7	269	199.5	329	247.0	389	296.8
150	108.8	210	154.5	270	200.3	330	247.7	390	297.7
151	109.6	211	155.2	271	201.1	331	248.5	391	298.5
152	110.3	212	156.0	272	201.9	332	249.2	392	299.4
153	111.1	213	156.7	273	202.7	333	250.0	393	300.3
154	111.9	214	157.5	274	203.5	334	250.8	394	301.1
155	112.6	215	158.2	275	204.3	335	251.6	395	302.0
156	113.4	216	159.0	276	205.1	336	252.5	396	302.8
157	114.1	217	159.7	277	205.9	337	253.3	397	303.7
158	114.9	218	160.4	278	206.7	338	254.1	398	304.6
159	115.6	219	161.2	279	207.5	339	254.9	399	305.4
								400	306.3

DEXTROSE.

50 *Approximate Volumetric Method for Rapid Work.—Tentative.*

Proceed as directed under 21. Standardize the reagent against pure dextrose.

51 *Soxhlet Method.—Tentative.*

Proceed as directed under 23. Under these conditions 100 cc. of the reagent require 0.475 gram of anhydrous dextrose for complete reduction and the formula becomes $\frac{100 \times 0.475}{VW} =$ per cent of dextrose.

52 *General Gravimetric Method.—Tentative.*

Proceed as directed under 25 and obtain, from 27, the weight of dextrose equivalent to the weight of copper reduced.

Allihn Gravimetric Method.—Tentative.

53

REAGENT.

Allihn's Modification of Fehling's Solution.—Prepare by mixing, immediately before use, equal volumes of (a) and (b).

(a) *Copper sulphate solution.*—Dissolve 34.639 grams of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water and dilute to 500 cc.

(b) *Alkaline tartrate solution.*—Dissolve 173 grams of Rochelle salts and 125 grams of potassium hydroxid in water and dilute to 500 cc.

54

DETERMINATION.

Place 30 cc. of the copper sulphate solution, 30 cc. of the alkaline tartrate solution, and 60 cc. of water in a beaker and heat to boiling. Add 25 cc. of the solution of the material to be examined, prepared so as not to contain more than 0.25 gram of dextrose, and boil for exactly 2 minutes, keeping the beaker covered. Filter immediately through asbestos, and obtain the weight of copper by one of the methods given under 26, 29–34 inclusive. The corresponding weight of dextrose is found in 55.

TABLE 7.—ALLIHN'S TABLE.¹⁰*For the determination of dextrose.*

[Expressed in milligrams.]

COPPER	CU- PROUS OXID	DEX- TROSE	COPPER	CU- PROUS OXID	DEX- TROSE	COPPER	CU- PROUS OXID	DEX- TROSE	COPPER	CU- PROUS OXID	DEX- TROSE
11	12.4	6.6	71	79.9	36.3	131	147.5	66.7	191	216.0	97.8
12	13.5	7.1	72	81.1	36.8	132	148.6	67.2	192	216.2	98.4
13	14.6	7.6	73	82.2	37.3	133	149.7	67.7	193	217.3	98.9
14	15.8	8.1	74	83.3	37.8	134	150.9	68.2	194	218.4	99.4
15	16.9	8.6	75	84.4	38.3	135	152.0	68.8	195	219.5	100.0
16	18.0	9.0	76	85.6	38.8	136	153.1	69.3	196	220.7	100.5
17	19.1	9.5	77	86.7	39.3	137	154.2	69.8	197	221.8	101.0
18	20.3	10.0	78	87.8	39.8	138	155.4	70.3	198	222.9	101.5
19	21.4	10.5	79	88.9	40.3	139	156.5	70.8	199	224.0	102.0
20	22.5	11.0	80	90.1	40.8	140	157.6	71.3	200	225.2	102.6
21	23.6	11.5	81	91.2	41.3	141	158.7	71.8	201	226.3	103.1
22	24.8	12.0	82	92.3	41.8	142	159.9	72.3	202	227.4	103.7
23	25.9	12.5	83	93.4	42.3	143	161.0	72.9	203	228.5	104.2
24	27.0	13.0	84	94.6	42.8	144	162.1	73.4	204	229.7	104.7
25	28.1	13.5	85	95.7	43.4	145	163.2	73.9	205	230.8	105.3
26	29.3	14.0	86	96.8	43.9	146	164.4	74.4	206	231.9	105.8
27	30.4	14.5	87	97.9	44.4	147	165.5	74.9	207	233.0	106.3
28	31.5	15.0	88	99.1	44.9	148	166.6	75.5	208	234.2	106.8
29	32.7	15.5	89	100.2	45.4	149	167.7	76.0	209	235.3	107.4
30	33.8	16.0	90	101.3	45.9	150	168.9	76.5	210	236.4	107.9
31	34.9	16.5	91	102.4	46.4	151	170.0	77.0	211	237.6	108.4
32	36.0	17.0	92	103.6	46.9	152	171.1	77.5	212	238.7	109.0
33	37.2	17.5	93	104.7	47.4	153	172.3	78.1	213	239.8	109.5
34	38.3	18.0	94	105.8	47.9	154	173.4	78.6	214	240.9	110.0
35	39.4	18.5	95	107.0	48.4	155	174.5	79.1	215	242.1	110.6
36	40.5	18.9	96	108.1	48.9	156	175.6	79.6	216	243.2	111.1
37	41.7	19.4	97	109.2	49.4	157	176.8	80.1	217	244.3	111.6
38	42.8	19.9	98	110.3	49.9	158	177.9	80.7	218	245.4	112.1
39	43.9	20.4	99	111.5	50.4	159	179.0	81.2	219	246.6	112.7
40	45.0	20.9	100	112.6	50.9	160	180.1	81.7	220	247.7	113.2
41	46.2	21.4	101	113.7	51.4	161	181.3	82.2	221	248.7	113.7
42	47.3	21.9	102	114.8	51.9	162	182.4	82.7	222	249.9	114.3
43	48.4	22.4	103	116.0	52.4	163	183.5	83.3	223	251.0	114.8
44	49.5	22.9	104	117.1	52.9	164	184.6	83.8	224	252.4	115.3
45	50.7	23.4	105	118.2	53.5	165	185.8	84.3	225	253.3	115.9
46	51.8	23.9	106	119.3	54.0	166	186.9	84.8	226	254.4	116.4
47	52.9	24.4	107	120.5	54.5	167	188.0	85.3	227	255.6	116.9
48	54.0	24.9	108	121.6	55.0	168	189.1	85.9	228	256.7	117.4
49	55.2	25.4	109	122.7	55.5	169	190.3	86.4	229	257.8	118.0
50	56.3	25.9	110	123.8	56.0	170	191.4	86.9	230	258.9	118.5
51	57.4	26.4	111	125.0	56.5	171	192.5	87.4	231	260.1	119.0
52	58.5	26.9	112	126.1	57.0	172	193.6	87.9	232	261.2	119.6
53	59.7	27.4	113	127.2	57.5	173	194.8	88.5	233	262.3	120.1
54	60.8	27.9	114	128.3	58.0	174	195.9	89.0	234	263.4	120.7
55	61.9	28.4	115	129.6	58.6	175	197.0	89.5	235	264.6	121.2
56	63.0	28.8	116	130.6	59.1	176	198.1	90.0	236	265.7	121.7
57	64.2	29.3	117	131.7	59.6	177	199.3	90.5	237	266.8	122.3
58	65.3	29.8	118	132.8	60.1	178	200.4	91.1	238	268.0	122.8
59	66.4	30.3	119	134.0	60.6	179	201.5	91.6	239	269.1	123.4
60	67.6	30.8	120	135.1	61.1	180	202.6	92.1	240	270.2	123.9
61	68.7	31.3	121	136.2	61.6	181	203.8	92.6	241	271.3	124.4
62	69.8	31.8	122	137.4	62.1	182	204.9	93.1	242	272.5	125.0
63	70.9	32.3	123	138.5	62.6	183	206.0	93.7	243	273.6	125.5
64	72.1	32.8	124	139.6	63.1	184	207.1	94.2	244	274.7	126.0
65	73.2	33.3	125	140.7	63.7	185	208.3	94.7	245	275.8	126.6
66	74.3	33.8	126	141.9	64.2	186	209.4	95.2	246	277.0	127.1
67	75.4	34.3	127	143.0	64.7	187	210.5	95.7	247	278.1	127.6
68	76.6	34.8	128	144.1	65.2	188	211.7	96.3	248	279.2	128.1
69	77.7	35.3	129	145.2	65.7	189	212.8	96.8	249	280.3	128.7
70	78.8	35.8	130	146.4	66.2	190	213.9	97.3	250	281.5	129.2

TABLE 7.—ALLIEN'S TABLE.—Continued.

[Expressed in milligrams.]

COPPER	CU- PROUS OXID	DEX- TROSE	COPPER	CU- PROUS OXID	DEX- TROSE	COPPER	CU- PROUS OXID	DEX- TROSE	COPPER	CU- PROUS OXID	DEX- TROSE
251	232.6	129.7	306	344.5	159.8	361	406.4	190.6	416	468.4	222.2
252	233.7	130.3	307	345.6	160.4	362	407.6	191.1	417	469.5	222.8
253	234.8	130.8	308	346.8	160.9	363	408.7	191.7	418	470.6	223.3
254	236.0	131.4	309	347.9	161.5	364	409.8	192.3	419	471.8	223.9
255	237.1	131.9	310	349.0	162.0	365	410.9	192.9	420	472.9	224.5
256	238.2	132.4	311	350.1	162.6	366	412.1	193.4	421	474.0	225.1
257	239.3	133.0	312	351.3	163.1	367	413.2	194.0	422	475.6	225.7
258	240.5	133.5	313	352.4	163.7	368	414.3	194.6	423	476.2	226.3
259	241.6	134.1	314	353.5	164.2	369	415.4	195.1	424	477.4	226.9
260	242.7	134.6	315	354.6	164.8	370	416.6	195.7	425	478.5	227.5
261	243.8	135.1	316	355.8	165.3	371	417.7	196.3	426	479.6	228.0
262	245.0	135.7	317	356.9	165.9	372	418.8	196.8	427	480.7	228.6
263	246.1	136.2	318	358.0	166.4	373	420.0	197.4	428	481.9	229.2
264	247.2	136.8	319	359.1	167.0	374	421.1	198.0	429	483.0	229.8
265	248.3	137.3	320	360.3	167.5	375	422.2	198.6	430	484.1	230.4
266	249.5	137.8	321	361.4	168.1	376	423.3	199.1	431	485.3	231.0
267	250.6	138.4	322	362.5	168.6	377	424.5	199.7	432	486.4	231.6
268	251.7	138.9	323	363.7	169.2	378	425.6	200.3	433	487.5	232.2
269	252.8	139.5	324	364.8	169.7	379	426.7	200.8	434	488.6	232.8
270	254.0	140.0	325	365.9	170.3	380	427.8	201.4	435	489.7	233.4
271	255.1	140.6	326	367.0	170.9	381	429.0	202.0	436	490.9	233.9
272	256.2	141.1	327	368.2	171.4	382	430.1	202.5	437	492.0	234.5
273	257.3	141.7	328	369.3	172.0	383	431.2	203.1	438	493.1	235.1
274	258.5	142.2	329	370.4	172.5	384	432.3	203.7	439	494.3	235.7
275	259.6	142.8	330	371.5	173.1	385	433.5	204.3	440	495.4	236.3
276	260.7	143.3	331	372.7	173.7	386	434.6	204.8	441	496.5	236.9
277	261.9	143.9	332	373.8	174.2	387	435.7	205.4	442	497.6	237.5
278	263.0	144.4	333	374.9	174.8	388	436.8	206.0	443	498.8	238.1
279	264.1	145.0	334	376.0	175.3	389	438.0	206.5	444	499.9	238.7
280	265.2	145.5	335	377.2	175.9	390	439.1	207.1	445	501.0	239.3
281	266.4	146.1	336	378.3	176.5	391	440.2	207.7	446	502.1	239.8
282	267.5	146.6	337	379.4	177.0	392	441.3	208.3	447	503.2	240.4
283	268.6	147.2	338	380.5	177.6	393	442.4	208.8	448	504.4	241.0
284	269.7	147.7	339	381.7	178.1	394	443.5	209.4	449	505.5	241.6
285	270.9	148.3	340	382.8	178.7	395	444.7	210.0	450	506.6	242.2
286	272.0	148.8	341	383.9	179.3	396	445.9	210.6	451	507.8	242.8
287	273.1	149.4	342	385.0	179.8	397	447.0	211.2	452	508.9	243.4
288	274.2	149.9	343	386.2	180.4	398	448.1	211.7	453	510.0	244.0
289	275.4	150.5	344	387.3	180.9	399	449.2	212.3	454	511.1	244.6
290	276.5	151.0	345	388.4	181.5	400	450.3	212.9	455	512.3	245.2
291	277.7	151.6	346	389.6	182.1	401	451.5	213.5	456	513.4	245.7
292	278.8	152.1	347	390.7	182.6	402	452.6	214.1	457	514.5	246.3
293	279.9	152.7	348	391.8	183.2	403	453.7	214.6	458	515.6	246.9
294	281.0	153.2	349	392.9	183.7	404	454.8	215.2	459	516.8	247.5
295	282.1	153.8	350	394.0	184.3	405	456.0	215.8	460	517.9	248.1
296	283.3	154.3	351	395.2	184.9	406	457.1	216.4	461	519.0	248.7
297	284.4	154.9	352	396.3	185.4	407	458.2	217.0	462	520.1	249.3
298	285.5	155.4	353	397.4	186.0	408	459.4	217.5	463	521.3	249.9
299	286.6	156.0	354	398.6	186.6	409	460.5	218.1			
300	287.8	156.5	355	399.7	187.2	410	461.6	218.7			
301	288.9	157.1	356	400.8	187.7	411	462.7	219.3			
302	290.0	157.6	357	401.9	188.3	412	463.8	219.9			
303	291.1	158.2	358	403.1	188.9	413	465.0	220.4			
304	292.3	158.7	359	404.2	189.4	414	466.1	221.0			
305	293.4	159.3	360	405.3	190.0	415	467.2	221.6			

56

REDUCING SUGARS OTHER THAN DEXTROSE.

Proceed as directed under 54 and multiply the weight of dextrose found in 55 by the following factors:

Levulose,	1.093;
Invert sugar,	1.046;
Arabinose,	0.969;
Xylose,	1.017;
Galactose,	1.114.

TOTAL SUGARS.¹¹

(Applicable to cattle foods.)

57

PREPARATION OF SOLUTION.

Place 12 grams of the material in a 300 cc. graduated flask, if the substance has an acid reaction add 1-3 grams of calcium carbonate, and boil on a steam bath for 1 hour with 150 cc. of 50% alcohol by volume, using a small funnel in the neck of the flask to condense the vapor. Cool, and allow the mixture to stand several hours, preferably overnight. Make up to volume with neutral 95% alcohol, mix thoroughly, allow to settle, transfer 200 cc. to a beaker with a pipette, and evaporate on a steam bath to a volume of 20-30 cc.

Do not evaporate to dryness, a little alcohol in the residue doing no harm. Transfer to a 100 cc. graduated flask, and rinse the beaker thoroughly with water, adding the rinsings to the contents of the flask. Add enough saturated neutral lead acetate solution to produce a flocculent precipitate, shake thoroughly and allow to stand 15 minutes. Make up to the mark with water, mix thoroughly, and filter through a dry filter. Add sufficient anhydrous sodium carbonate to the filtrate to precipitate all the lead, again filter through a dry paper and test the filtrate with a little anhydrous sodium carbonate to make sure that all the lead has been removed.

58

DETERMINATION OF REDUCING SUGARS.

Proceed as directed under 26 or 29-34 respectively, employing the Soxhlet modification of Fehling's solution and using 25 cc. of the solution (representing 2 grams of the sample), prepared as directed in 57. Express the results as dextrose or invert sugar.

59

SUCROSE.

Introduce 50 cc. of the solution, prepared as directed in 57, into a 100 cc. graduated flask, add a piece of litmus paper, neutralize with acetic acid, add 5 cc. of concentrated hydrochloric acid and allow the inversion to proceed at room temperature as directed under 14 or 16. When inversion is complete, transfer the solution to a beaker, neutralize with sodium carbonate, return the solution to the 100 cc. flask, dilute to the mark with water, filter if necessary and determine reducing sugars in 50 cc. of the solution (representing 2 grams of the sample) as directed in 58, and calculate the results as invert sugar. Subtract the per cent of reducing sugars before inversion from the per cent of total sugar after inversion, both calculated as invert sugar, and multiply the difference by 0.95 to obtain the per cent of sucrose present.

Since the insoluble material of grain or cattle food occupies some space in the flask as originally made up, it is necessary to correct for this volume. Results of a large number of determinations on various materials have shown the average volume of 12 grams of material to be 9 cc., and therefore to obtain the true amount of sugars present all results must be multiplied by the factor 0.97.

STARCH.

60

Direct Acid Hydrolysis (Modified Sachsse Method).—Official.

(In this method there will be included as starch the pentosans and other carbohydrate bodies present which undergo hydrolysis and conversion into reducing sugars on boiling with hydrochloric acid.)

Stir a quantity of the sample, representing 2.5–3 grams of the dry material, in a beaker with 50 cc. of cold water for an hour. Transfer to a filter and wash with 250 cc. of cold water. Heat the insoluble residue for 2½ hours with 200 cc. of water and 20 cc. of hydrochloric acid (sp. gr. 1.125) in a flask provided with a reflux condenser. Cool, and nearly neutralize with sodium hydroxid. Complete the volume to 250 cc., filter, and determine the dextrose in an aliquot of the filtrate as directed under 52 or 54. The weight of the dextrose obtained multiplied by 0.90 gives the weight of starch.

The factor 0.9 is the theoretical ratio between starch and glucose but, according to Noyes¹² and other investigators, the factor 0.93 more nearly approaches the actual yield.

Diastase Method with Subsequent Acid Hydrolysis.—Tentative.

61

REAGENT.

Malt extract.—Digest 10 grams of fresh, finely ground malt for 2–3 hours at ordinary temperature with 200 cc. of water and filter. Determine the amount of dextrose in a given quantity of the filtrate after boiling with acid, etc., as in the starch determination, and make the proper correction in the subsequent determination.

62

DETERMINATION.

Extract a convenient quantity of the substance (ground to an impalpable powder and representing 4–5 grams of the dry material) on a hardened filter with 5 successive portions of 10 cc. of ether; wash with 150 cc. of 10% alcohol and then with a little strong alcohol. Place the residue in a beaker with 50 cc. of water, immerse the beaker in boiling water, and stir constantly for 15 minutes or until all the starch is gelatinized; cool to 55°C., add 20 cc. of malt extract, and maintain at this temperature for an hour. Heat again to boiling for a few minutes, cool to 55°C., add 20 cc. of malt extract, and maintain at this temperature for an hour or until the residue treated with iodine shows no blue color upon microscopic examination. Cool, make up directly to 250 cc., and filter. Place 200 cc. of the filtrate in a flask with 20 cc. of hydrochloric acid (sp. gr. 1.125); connect with a reflux condenser and heat in a boiling water bath for 2½ hours. Cool, nearly neutralize with sodium hydroxid solution, finish the neutralization with sodium carbonate solution, and make up to 500 cc. Mix the solution well, pour through a dry filter, and determine the dextrose in an aliquot as directed under 52 or 54. Conduct a blank determination upon the same volume of the malt extract as used upon the sample and correct the weight of reduced copper accordingly. The weight of the dextrose obtained multiplied by 0.90 gives the weight of starch.

PENTOSANS.—TENTATIVE.

63

REAGENT.

Phloroglucin.—Dissolve a small quantity of the phloroglucin in a few drops of acetic anhydride, heat almost to boiling, and add a few drops of concentrated sul-

phuric acid. A violet color indicates the presence of diresorcin. A phloroglucin which gives more than a faint coloration may be purified by the following method:

Heat in a beaker about 300 cc. of hydrochloric acid (sp. gr. 1.06) and 11 grams of commercial phloroglucin, added in small quantities at a time, stirring constantly until it has almost entirely dissolved. Pour the hot solution into a sufficient quantity of the same hydrochloric acid (cold) to make the volume 1500 cc. Allow it to stand at least overnight, preferably several days, to permit the diresorcin to crystallize out. Filter immediately before using. A yellow tint does not interfere with its usefulness. In using it, add the volume containing the required amount to the distillate.

64

DETERMINATION.

Place a quantity of the material, 2-5 grams, chosen so that the weight of phloroglucid obtained shall not exceed 0.300 gram, in a 300 cc. distillation flask, together with 100 cc. of 12% hydrochloric acid (sp. gr. 1.06), and several pieces of recently heated pumice stone. Place the flask on a wire gauze, connect with a condenser, and heat, rather gently at first, and regulate so as to distil over 30 cc. in about 10 minutes, the distillate passing through a small filter paper. Replace the 30 cc. distilled by a like quantity of the dilute acid, added by means of a separatory funnel in such a manner as to wash down the particles adhering to the sides of the flask, and continue the process until the distillate amounts to 360 cc. To the total distillate add gradually a quantity of phloroglucin dissolved in 12% hydrochloric acid and stir thoroughly the resulting mixture. The amount of phloroglucin used should be about double that of the furfural expected. The solution turns first yellow, then green, and very soon an amorphous greenish precipitate appears, which grows darker rapidly, till it becomes finally almost black. Make the solution up to 400 cc. with 12% hydrochloric acid, and allow to stand overnight.

Filter the amorphous black precipitate into a tared Gooch crucible through an asbestos mat, wash carefully with 150 cc. of water in such a way that the water is not entirely removed from the crucible until the very last, then dry for 4 hours at the temperature of boiling water, cool and weigh in a weighing bottle, the increase in weight being reckoned as furfural phloroglucid. To calculate the furfural, pentose, or pentosan from the phloroglucid, use the following formulas given by Kröber:

(1) For a weight of phloroglucid, designated by "a" in the following formulas, under 0.03 gram,

$$\text{Furfural} = (a + 0.0052) \times 0.5170.$$

$$\text{Pentoses} = (a + 0.0052) \times 1.0170.$$

$$\text{Pentosans} = (a + 0.0052) \times 0.8949.$$

In the above and also in the following formulas, the factor 0.0052 represents the weight of phloroglucid which remains dissolved in the 400 cc. of acid solution.

(2) For a weight of phloroglucid "a" over 0.300 gram,

$$\text{Furfural} = (a + 0.0052) \times 0.5180.$$

$$\text{Pentoses} = (a + 0.0052) \times 1.0026.$$

$$\text{Pentosans} = (a + 0.0052) \times 0.8824.$$

For a weight of phloroglucid "a" between 0.03 and 0.300 gram use Kröber's table, 65, or the following formulas in which the factors were calculated from Kröber's tables by C. A. Browne,¹³

$$\text{Furfural} = (a + 0.0052) \times 0.5185.$$

$$\text{Pentoses} = (a + 0.0052) \times 1.0075.$$

$$\text{Pentosans} = (a + 0.0052) \times 0.8866.$$

TABLE 8.—KRÖBER'S TABLE.¹⁴
For Determining Pentoses and Pentosans.
 [Expressed in grams.]

FURFURAL PHENOLGLUCID	FURFURAL	ARABINOSE	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.030	0.0182	0.0391	0.0344	0.0324	0.0285	0.0358	0.0315
0.031	0.0188	0.0402	0.0354	0.0333	0.0293	0.0368	0.0324
0.032	0.0193	0.0413	0.0363	0.0342	0.0301	0.0378	0.0333
0.033	0.0198	0.0424	0.0373	0.0352	0.0309	0.0388	0.0341
0.034	0.0203	0.0435	0.0383	0.0361	0.0317	0.0398	0.0350
0.035	0.0209	0.0446	0.0393	0.0370	0.0326	0.0408	0.0359
0.036	0.0214	0.0457	0.0402	0.0379	0.0334	0.0418	0.0368
0.037	0.0219	0.0468	0.0412	0.0388	0.0342	0.0428	0.0377
0.038	0.0224	0.0479	0.0422	0.0398	0.0350	0.0439	0.0386
0.039	0.0229	0.0490	0.0431	0.0407	0.0358	0.0449	0.0395
0.040	0.0235	0.0501	0.0441	0.0416	0.0366	0.0459	0.0404
0.041	0.0240	0.0512	0.0451	0.0425	0.0374	0.0469	0.0413
0.042	0.0245	0.0523	0.0460	0.0434	0.0382	0.0479	0.0422
0.043	0.0250	0.0534	0.0470	0.0443	0.0390	0.0489	0.0431
0.044	0.0255	0.0545	0.0480	0.0452	0.0398	0.0499	0.0440
0.045	0.0260	0.0556	0.0490	0.0462	0.0406	0.0509	0.0448
0.046	0.0266	0.0567	0.0499	0.0471	0.0414	0.0519	0.0457
0.047	0.0271	0.0578	0.0509	0.0480	0.0422	0.0529	0.0466
0.048	0.0276	0.0589	0.0519	0.0489	0.0430	0.0539	0.0475
0.049	0.0281	0.0600	0.0528	0.0498	0.0438	0.0549	0.0484
0.050	0.0286	0.0611	0.0538	0.0507	0.0446	0.0559	0.0492
0.051	0.0292	0.0622	0.0548	0.0516	0.0454	0.0569	0.0501
0.052	0.0297	0.0633	0.0557	0.0525	0.0462	0.0579	0.0510
0.053	0.0302	0.0644	0.0567	0.0534	0.0470	0.0589	0.0519
0.054	0.0307	0.0655	0.0576	0.0543	0.0478	0.0599	0.0528
0.055	0.0312	0.0666	0.0586	0.0553	0.0486	0.0610	0.0537
0.056	0.0318	0.0677	0.0596	0.0562	0.0494	0.0620	0.0546
0.057	0.0323	0.0688	0.0605	0.0571	0.0502	0.0630	0.0555
0.058	0.0328	0.0699	0.0615	0.0580	0.0510	0.0640	0.0564
0.059	0.0333	0.0710	0.0624	0.0589	0.0518	0.0650	0.0573
0.060	0.0338	0.0721	0.0634	0.0598	0.0526	0.0660	0.0581
0.061	0.0344	0.0732	0.0644	0.0607	0.0534	0.0670	0.0590
0.062	0.0349	0.0743	0.0653	0.0616	0.0542	0.0680	0.0599
0.063	0.0354	0.0754	0.0663	0.0626	0.0550	0.0690	0.0608
0.064	0.0359	0.0765	0.0673	0.0635	0.0558	0.0700	0.0617
0.065	0.0364	0.0776	0.0683	0.0644	0.0567	0.0710	0.0625
0.066	0.0370	0.0787	0.0692	0.0653	0.0575	0.0720	0.0634
0.067	0.0375	0.0798	0.0702	0.0662	0.0583	0.0730	0.0643
0.068	0.0380	0.0809	0.0712	0.0672	0.0591	0.0741	0.0652
0.069	0.0385	0.0820	0.0721	0.0681	0.0599	0.0751	0.0661
0.070	0.0390	0.0831	0.0731	0.0690	0.0607	0.0761	0.0670
0.071	0.0396	0.0842	0.0741	0.0699	0.0615	0.0771	0.0679
0.072	0.0401	0.0853	0.0750	0.0708	0.0623	0.0781	0.0688
0.073	0.0406	0.0864	0.0760	0.0717	0.0631	0.0791	0.0697
0.074	0.0411	0.0875	0.0770	0.0726	0.0639	0.0801	0.0706

TABLE 8.—KRÖBER'S TABLE.—Continued.

[Expressed in grams.]

FURFURAL FELOROGUCID	FURFURAL	ARABINOS	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.075	0.0416	0.0886	0.0780	0.0736	0.0647	0.0811	0.0714
0.076	0.0422	0.0897	0.0789	0.0745	0.0655	0.0821	0.0722
0.077	0.0427	0.0908	0.0799	0.0754	0.0663	0.0831	0.0731
0.078	0.0432	0.0919	0.0809	0.0763	0.0671	0.0841	0.0740
0.079	0.0437	0.0930	0.0818	0.0772	0.0679	0.0851	0.0749
0.080	0.0442	0.0941	0.0828	0.0781	0.0687	0.0861	0.0758
0.081	0.0448	0.0952	0.0838	0.0790	0.0695	0.0871	0.0767
0.082	0.0453	0.0963	0.0847	0.0799	0.0703	0.0881	0.0776
0.083	0.0458	0.0974	0.0857	0.0808	0.0711	0.0891	0.0785
0.084	0.0463	0.0985	0.0867	0.0817	0.0719	0.0901	0.0794
0.085	0.0468	0.0996	0.0877	0.0827	0.0727	0.0912	0.0803
0.086	0.0474	0.1007	0.0886	0.0836	0.0735	0.0922	0.0812
0.087	0.0479	0.1018	0.0896	0.0845	0.0743	0.0932	0.0821
0.088	0.0484	0.1029	0.0906	0.0854	0.0751	0.0942	0.0830
0.089	0.0489	0.1040	0.0915	0.0863	0.0759	0.0952	0.0838
0.090	0.0494	0.1051	0.0925	0.0872	0.0767	0.0962	0.0847
0.091	0.0499	0.1062	0.0935	0.0881	0.0775	0.0972	0.0856
0.092	0.0505	0.1073	0.0944	0.0890	0.0783	0.0982	0.0865
0.093	0.0510	0.1084	0.0954	0.0900	0.0791	0.0992	0.0874
0.094	0.0515	0.1095	0.0964	0.0909	0.0800	0.1002	0.0883
0.095	0.0520	0.1106	0.0974	0.0918	0.0808	0.1012	0.0891
0.096	0.0525	0.1117	0.0983	0.0927	0.0816	0.1022	0.0899
0.097	0.0531	0.1128	0.0993	0.0936	0.0824	0.1032	0.0908
0.098	0.0536	0.1139	0.1003	0.0946	0.0832	0.1043	0.0917
0.099	0.0541	0.1150	0.1012	0.0955	0.0840	0.1053	0.0926
0.100	0.0546	0.1161	0.1022	0.0964	0.0848	0.1063	0.0935
0.101	0.0551	0.1171	0.1032	0.0973	0.0856	0.1073	0.0944
0.102	0.0557	0.1182	0.1041	0.0982	0.0864	0.1083	0.0953
0.103	0.0562	0.1193	0.1051	0.0991	0.0872	0.1093	0.0962
0.104	0.0567	0.1204	0.1060	0.1000	0.0880	0.1103	0.0971
0.105	0.0572	0.1215	0.1070	0.1010	0.0888	0.1113	0.0979
0.106	0.0577	0.1226	0.1080	0.1019	0.0896	0.1123	0.0988
0.107	0.0582	0.1237	0.1089	0.1028	0.0904	0.1133	0.0997
0.108	0.0588	0.1248	0.1099	0.1037	0.0912	0.1143	0.1006
0.109	0.0593	0.1259	0.1108	0.1046	0.0920	0.1153	0.1015
0.110	0.0598	0.1270	0.1118	0.1055	0.0928	0.1163	0.1023
0.111	0.0603	0.1281	0.1128	0.1064	0.0936	0.1173	0.1032
0.112	0.0608	0.1292	0.1137	0.1073	0.0944	0.1183	0.1041
0.113	0.0614	0.1303	0.1147	0.1082	0.0952	0.1193	0.1050
0.114	0.0619	0.1314	0.1156	0.1091	0.0960	0.1203	0.1059
0.115	0.0624	0.1325	0.1166	0.1101	0.0968	0.1213	0.1067
0.116	0.0629	0.1336	0.1176	0.1110	0.0976	0.1223	0.1076
0.117	0.0634	0.1347	0.1185	0.1119	0.0984	0.1233	0.1085
0.118	0.0640	0.1358	0.1195	0.1128	0.0992	0.1243	0.1094
0.119	0.0645	0.1369	0.1204	0.1137	0.1000	0.1253	0.1103

TABLE 8.—KRÖBER'S TABLE.—Continued.

[Expressed in grams.]

FURFURAL PELOROGUCID	FURFURAL	ARABINOSE	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.120	0.0650	0.1380	0.1214	0.1146	0.1008	0.1263	0.1111
0.121	0.0655	0.1391	0.1224	0.1155	0.1016	0.1273	0.1120
0.122	0.0660	0.1402	0.1233	0.1164	0.1024	0.1283	0.1129
0.123	0.0665	0.1413	0.1243	0.1173	0.1032	0.1293	0.1138
0.124	0.0671	0.1424	0.1253	0.1182	0.1040	0.1303	0.1147
0.125	0.0676	0.1435	0.1263	0.1192	0.1049	0.1314	0.1156
0.126	0.0681	0.1446	0.1272	0.1201	0.1057	0.1324	0.1165
0.127	0.0686	0.1457	0.1282	0.1210	0.1065	0.1334	0.1174
0.128	0.0691	0.1468	0.1292	0.1219	0.1073	0.1344	0.1183
0.129	0.0697	0.1479	0.1301	0.1228	0.1081	0.1354	0.1192
0.130	0.0702	0.1490	0.1311	0.1237	0.1089	0.1364	0.1201
0.131	0.0707	0.1501	0.1321	0.1246	0.1097	0.1374	0.1210
0.132	0.0712	0.1512	0.1330	0.1255	0.1105	0.1384	0.1219
0.133	0.0717	0.1523	0.1340	0.1264	0.1113	0.1394	0.1227
0.134	0.0723	0.1534	0.1350	0.1273	0.1121	0.1404	0.1236
0.135	0.0728	0.1545	0.1360	0.1283	0.1129	0.1414	0.1244
0.136	0.0733	0.1556	0.1369	0.1292	0.1137	0.1424	0.1253
0.137	0.0738	0.1567	0.1379	0.1301	0.1145	0.1434	0.1262
0.138	0.0743	0.1578	0.1389	0.1310	0.1153	0.1444	0.1271
0.139	0.0748	0.1589	0.1398	0.1319	0.1161	0.1454	0.1280
0.140	0.0754	0.1600	0.1408	0.1328	0.1169	0.1464	0.1288
0.141	0.0759	0.1611	0.1418	0.1337	0.1177	0.1474	0.1297
0.142	0.0764	0.1622	0.1427	0.1346	0.1185	0.1484	0.1306
0.143	0.0769	0.1633	0.1437	0.1355	0.1193	0.1494	0.1315
0.144	0.0774	0.1644	0.1447	0.1364	0.1201	0.1504	0.1324
0.145	0.0780	0.1655	0.1457	0.1374	0.1209	0.1515	0.1333
0.146	0.0785	0.1666	0.1466	0.1383	0.1217	0.1525	0.1342
0.147	0.0790	0.1677	0.1476	0.1392	0.1225	0.1535	0.1351
0.148	0.0795	0.1688	0.1486	0.1401	0.1233	0.1545	0.1360
0.149	0.0800	0.1699	0.1495	0.1410	0.1241	0.1555	0.1369
0.150	0.0805	0.1710	0.1505	0.1419	0.1249	0.1565	0.1377
0.151	0.0811	0.1721	0.1515	0.1428	0.1257	0.1575	0.1386
0.152	0.0816	0.1732	0.1524	0.1437	0.1265	0.1585	0.1395
0.153	0.0821	0.1743	0.1534	0.1446	0.1273	0.1595	0.1404
0.154	0.0826	0.1754	0.1544	0.1455	0.1281	0.1605	0.1413
0.155	0.0831	0.1765	0.1554	0.1465	0.1289	0.1615	0.1421
0.156	0.0837	0.1776	0.1563	0.1474	0.1297	0.1625	0.1430
0.157	0.0842	0.1787	0.1573	0.1483	0.1305	0.1635	0.1439
0.158	0.0847	0.1798	0.1583	0.1492	0.1313	0.1645	0.1448
0.159	0.0852	0.1809	0.1592	0.1501	0.1321	0.1655	0.1457
0.160	0.0857	0.1820	0.1602	0.1510	0.1329	0.1665	0.1465
0.161	0.0863	0.1831	0.1612	0.1519	0.1337	0.1675	0.1474
0.162	0.0868	0.1842	0.1621	0.1528	0.1345	0.1685	0.1483
0.163	0.0873	0.1853	0.1631	0.1537	0.1353	0.1695	0.1492
0.164	0.0878	0.1864	0.1640	0.1546	0.1361	0.1705	0.1501

TABLE 8.—KRÖBER'S TABLE.—Continued.

[Expressed in grams.]

FURFURAL FELOROGUCID	FURFURAL	ARABINOSE	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.165	0.0883	0.1875	0.1650	0.1556	0.1369	0.1716	0.1510
0.166	0.0888	0.1886	0.1660	0.1565	0.1377	0.1726	0.1519
0.167	0.0894	0.1897	0.1669	0.1574	0.1385	0.1736	0.1528
0.168	0.0899	0.1908	0.1679	0.1583	0.1393	0.1746	0.1537
0.169	0.0904	0.1919	0.1688	0.1592	0.1401	0.1756	0.1546
0.170	0.0909	0.1930	0.1698	0.1601	0.1409	0.1766	0.1554
0.171	0.0914	0.1941	0.1708	0.1610	0.1417	0.1776	0.1563
0.172	0.0920	0.1952	0.1717	0.1619	0.1425	0.1786	0.1572
0.173	0.0925	0.1963	0.1727	0.1628	0.1433	0.1796	0.1581
0.174	0.0930	0.1974	0.1736	0.1637	0.1441	0.1806	0.1590
0.175	0.0935	0.1985	0.1746	0.1647	0.1449	0.1816	0.1598
0.176	0.0940	0.1996	0.1756	0.1656	0.1457	0.1826	0.1607
0.177	0.0946	0.2007	0.1765	0.1665	0.1465	0.1836	0.1616
0.178	0.0951	0.2018	0.1775	0.1674	0.1473	0.1846	0.1625
0.179	0.0956	0.2029	0.1784	0.1683	0.1481	0.1856	0.1634
0.180	0.0961	0.2039	0.1794	0.1692	0.1489	0.1866	0.1642
0.181	0.0966	0.2050	0.1804	0.1701	0.1497	0.1876	0.1651
0.182	0.0971	0.2061	0.1813	0.1710	0.1505	0.1886	0.1660
0.183	0.0977	0.2072	0.1823	0.1719	0.1513	0.1896	0.1669
0.184	0.0982	0.2082	0.1832	0.1728	0.1521	0.1906	0.1678
0.185	0.0987	0.2093	0.1842	0.1738	0.1529	0.1916	0.1686
0.186	0.0992	0.2104	0.1851	0.1747	0.1537	0.1926	0.1695
0.187	0.0997	0.2115	0.1861	0.1756	0.1545	0.1936	0.1704
0.188	0.1003	0.2126	0.1870	0.1765	0.1553	0.1946	0.1712
0.189	0.1008	0.2136	0.1880	0.1774	0.1561	0.1955	0.1721
0.190	0.1013	0.2147	0.1889	0.1783	0.1569	0.1965	0.1729
0.191	0.1018	0.2158	0.1899	0.1792	0.1577	0.1975	0.1738
0.192	0.1023	0.2168	0.1908	0.1801	0.1585	0.1985	0.1747
0.193	0.1028	0.2179	0.1918	0.1810	0.1593	0.1995	0.1756
0.194	0.1034	0.2190	0.1927	0.1819	0.1601	0.2005	0.1764
0.195	0.1039	0.2201	0.1937	0.1829	0.1609	0.2015	0.1773
0.196	0.1044	0.2212	0.1946	0.1838	0.1617	0.2025	0.1782
0.197	0.1049	0.2222	0.1956	0.1847	0.1625	0.2035	0.1791
0.198	0.1054	0.2233	0.1965	0.1856	0.1633	0.2045	0.1800
0.199	0.1059	0.2244	0.1975	0.1865	0.1641	0.2055	0.1808
0.200	0.1065	0.2255	0.1984	0.1874	0.1649	0.2065	0.1817
0.201	0.1070	0.2266	0.1994	0.1883	0.1657	0.2075	0.1826
0.202	0.1075	0.2276	0.2003	0.1892	0.1665	0.2085	0.1835
0.203	0.1080	0.2287	0.2013	0.1901	0.1673	0.2095	0.1844
0.204	0.1085	0.2298	0.2022	0.1910	0.1681	0.2105	0.1853
0.205	0.1090	0.2309	0.2032	0.1920	0.1689	0.2115	0.1861
0.206	0.1096	0.2320	0.2041	0.1929	0.1697	0.2125	0.1869
0.207	0.1101	0.2330	0.2051	0.1938	0.1705	0.2134	0.1878
0.208	0.1106	0.2341	0.2060	0.1947	0.1713	0.2144	0.1887
0.209	0.1111	0.2352	0.2069	0.1956	0.1721	0.2154	0.1896

TABLE 8.—KRÖBER'S TABLE.—Continued.

[Expressed in grams.]

FURFURAL PHLOROGLUCID	FURFURAL	ARABINOSE	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.210	0.1116	0.2363	0.2079	0.1965	0.1729	0.2164	0.1904
0.211	0.1121	0.2374	0.2089	0.1975	0.1737	0.2174	0.1913
0.212	0.1127	0.2384	0.2098	0.1984	0.1745	0.2184	0.1922
0.213	0.1132	0.2395	0.2108	0.1993	0.1753	0.2194	0.1931
0.214	0.1137	0.2406	0.2117	0.2002	0.1761	0.2204	0.1940
0.215	0.1142	0.2417	0.2127	0.2011	0.1770	0.2214	0.1948
0.216	0.1147	0.2428	0.2136	0.2020	0.1778	0.2224	0.1957
0.217	0.1152	0.2438	0.2146	0.2029	0.1786	0.2234	0.1966
0.218	0.1158	0.2449	0.2155	0.2038	0.1794	0.2244	0.1974
0.219	0.1163	0.2460	0.2165	0.2047	0.1802	0.2254	0.1983
0.220	0.1168	0.2471	0.2174	0.2057	0.1810	0.2264	0.1992
0.221	0.1173	0.2482	0.2184	0.2066	0.1818	0.2274	0.2001
0.222	0.1178	0.2492	0.2193	0.2075	0.1826	0.2284	0.2010
0.223	0.1183	0.2503	0.2203	0.2084	0.1834	0.2294	0.2019
0.224	0.1189	0.2514	0.2212	0.2093	0.1842	0.2304	0.2028
0.225	0.1194	0.2525	0.2222	0.2102	0.1850	0.2314	0.2037
0.226	0.1199	0.2536	0.2232	0.2111	0.1858	0.2324	0.2046
0.227	0.1204	0.2546	0.2241	0.2121	0.1866	0.2334	0.2054
0.228	0.1209	0.2557	0.2251	0.2130	0.1874	0.2344	0.2063
0.229	0.1214	0.2568	0.2260	0.2139	0.1882	0.2354	0.2072
0.230	0.1220	0.2579	0.2270	0.2148	0.1890	0.2364	0.2081
0.231	0.1225	0.2590	0.2280	0.2157	0.1898	0.2374	0.2089
0.232	0.1230	0.2600	0.2289	0.2166	0.1906	0.2383	0.2097
0.233	0.1235	0.2611	0.2299	0.2175	0.1914	0.2393	0.2106
0.234	0.1240	0.2622	0.2308	0.2184	0.1922	0.2403	0.2115
0.235	0.1245	0.2633	0.2318	0.2193	0.1930	0.2413	0.2124
0.236	0.1251	0.2644	0.2327	0.2202	0.1938	0.2423	0.2132
0.237	0.1256	0.2654	0.2337	0.2211	0.1946	0.2433	0.2141
0.238	0.1261	0.2665	0.2346	0.2220	0.1954	0.2443	0.2150
0.239	0.1266	0.2676	0.2356	0.2229	0.1962	0.2453	0.2159
0.240	0.1271	0.2687	0.2365	0.2239	0.1970	0.2463	0.2168
0.241	0.1276	0.2698	0.2375	0.2248	0.1978	0.2473	0.2176
0.242	0.1281	0.2708	0.2384	0.2257	0.1986	0.2483	0.2185
0.243	0.1287	0.2719	0.2394	0.2266	0.1994	0.2493	0.2194
0.244	0.1292	0.2730	0.2403	0.2275	0.2002	0.2503	0.2203
0.245	0.1297	0.2741	0.2413	0.2284	0.2010	0.2513	0.2212
0.246	0.1302	0.2752	0.2422	0.2293	0.2018	0.2523	0.2220
0.247	0.1307	0.2762	0.2432	0.2302	0.2026	0.2533	0.2229
0.248	0.1312	0.2773	0.2441	0.2311	0.2034	0.2543	0.2238
0.249	0.1318	0.2784	0.2451	0.2320	0.2042	0.2553	0.2247
0.250	0.1323	0.2795	0.2460	0.2330	0.2050	0.2563	0.2256
0.251	0.1328	0.2806	0.2470	0.2339	0.2058	0.2573	0.2264
0.252	0.1333	0.2816	0.2479	0.2348	0.2066	0.2582	0.2272
0.253	0.1338	0.2827	0.2489	0.2357	0.2074	0.2592	0.2281
0.254	0.1343	0.2838	0.2498	0.2366	0.2082	0.2602	0.2290

TABLE 8.—KRÖBER'S TABLE.—Continued.

[Expressed in grams.]

FURFURAL PHLOROGLUCID	FURFURAL	ARABINOS	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.255	0.1349	0.2849	0.2508	0.2375	0.2090	0.2612	0.2299
0.256	0.1354	0.2860	0.2517	0.2384	0.2098	0.2622	0.2307
0.257	0.1359	0.2870	0.2526	0.2393	0.2106	0.2632	0.2316
0.258	0.1364	0.2881	0.2536	0.2402	0.2114	0.2642	0.2325
0.259	0.1369	0.2892	0.2545	0.2411	0.2122	0.2652	0.2334
0.260	0.1374	0.2903	0.2555	0.2420	0.2130	0.2662	0.2342
0.261	0.1380	0.2914	0.2565	0.2429	0.2138	0.2672	0.2351
0.262	0.1385	0.2924	0.2574	0.2438	0.2146	0.2681	0.2359
0.263	0.1390	0.2935	0.2584	0.2447	0.2154	0.2691	0.2368
0.264	0.1395	0.2946	0.2593	0.2456	0.2162	0.2701	0.2377
0.265	0.1400	0.2957	0.2603	0.2465	0.2170	0.2711	0.2385
0.266	0.1405	0.2968	0.2612	0.2474	0.2178	0.2721	0.2394
0.267	0.1411	0.2978	0.2622	0.2483	0.2186	0.2731	0.2403
0.268	0.1416	0.2989	0.2631	0.2492	0.2194	0.2741	0.2412
0.269	0.1421	0.3000	0.2641	0.2502	0.2202	0.2751	0.2421
0.270	0.1426	0.3011	0.2650	0.2511	0.2210	0.2761	0.2429
0.271	0.1431	0.3022	0.2660	0.2520	0.2218	0.2771	0.2438
0.272	0.1436	0.3032	0.2669	0.2529	0.2226	0.2781	0.2447
0.273	0.1442	0.3043	0.2679	0.2538	0.2234	0.2791	0.2456
0.274	0.1447	0.3054	0.2688	0.2547	0.2242	0.2801	0.2465
0.275	0.1452	0.3065	0.2698	0.2556	0.2250	0.2811	0.2473
0.276	0.1457	0.3076	0.2707	0.2565	0.2258	0.2821	0.2482
0.277	0.1462	0.3086	0.2717	0.2574	0.2266	0.2830	0.2490
0.278	0.1467	0.3097	0.2726	0.2583	0.2274	0.2840	0.2499
0.279	0.1473	0.3108	0.2736	0.2592	0.2282	0.2850	0.2508
0.280	0.1478	0.3119	0.2745	0.2602	0.2290	0.2861	0.2517
0.281	0.1483	0.3130	0.2755	0.2611	0.2298	0.2871	0.2526
0.282	0.1488	0.3140	0.2764	0.2620	0.2306	0.2880	0.2534
0.283	0.1493	0.3151	0.2774	0.2629	0.2314	0.2890	0.2543
0.284	0.1498	0.3162	0.2783	0.2638	0.2322	0.2900	0.2552
0.285	0.1504	0.3173	0.2793	0.2647	0.2330	0.2910	0.2561
0.286	0.1509	0.3184	0.2802	0.2656	0.2338	0.2920	0.2570
0.287	0.1514	0.3194	0.2812	0.2665	0.2346	0.2930	0.2578
0.288	0.1519	0.3205	0.2821	0.2674	0.2354	0.2940	0.2587
0.289	0.1524	0.3216	0.2831	0.2683	0.2362	0.2950	0.2596
0.290	0.1529	0.3227	0.2840	0.2693	0.2370	0.2960	0.2605
0.291	0.1535	0.3238	0.2850	0.2702	0.2378	0.2970	0.2614
0.292	0.1540	0.3248	0.2859	0.2711	0.2386	0.2980	0.2622
0.293	0.1545	0.3259	0.2868	0.2720	0.2394	0.2990	0.2631
0.294	0.1550	0.3270	0.2878	0.2729	0.2402	0.3000	0.2640
0.295	0.1555	0.3281	0.2887	0.2738	0.2410	0.3010	0.2649
0.296	0.1560	0.3292	0.2897	0.2747	0.2418	0.3020	0.2658
0.297	0.1566	0.3302	0.2906	0.2756	0.2426	0.3030	0.2666
0.298	0.1571	0.3313	0.2916	0.2765	0.2434	0.3040	0.2675
0.299	0.1576	0.3324	0.2925	0.2774	0.2442	0.3050	0.2684
0.300	0.1581	0.3335	0.2935	0.2784	0.2450	0.3060	0.2693

66

GALACTAN.—TENTATIVE.

Extract a convenient quantity of the substance, representing 2.5–3 grams of the dry material, on a hardened filter with 5 successive portions of 10 cc. of ether, place the extracted residue in a beaker, about 5.5 cm. in diameter and 7 cm. deep, together with 60 cc. of nitric acid of 1.15 sp. gr., and evaporate the solution to exactly one third its volume in a water bath at a temperature of 94°–96°C. After standing 24 hours, add 10 cc. of water to the precipitate, and allow it to stand another 24 hours. The mucic acid has in the meantime crystallized, but it is mixed with considerable material only partially oxidized by the nitric acid. Filter the solution through filter paper, wash with 30 cc. of water to remove as much of the nitric acid as possible, and replace the filter and contents in the beaker. Add 30 cc. of ammonium carbonate solution, consisting of 1 part ammonium carbonate, 19 parts water, and 1 part strong ammonium hydroxid, and heat the mixture on a water bath, at 80°C., for 15 minutes, with constant stirring. The ammonium carbonate takes up the mucic acid, forming soluble ammonium mucate. Wash the filter paper and contents several times with hot water by decantation, passing the washings through a filter paper, to which finally transfer the material and thoroughly wash. Evaporate the filtrate to dryness over a water bath, avoiding unnecessary heating which causes decomposition, add 5 cc. of nitric acid of 1.15 sp. gr., stir thoroughly the mixture and allow to stand for 30 minutes. The nitric acid decomposes the ammonium mucate, precipitating the mucic acid; collect this on a tared filter or Gooch, wash with 10–15 cc. of water, then with 60 cc. of alcohol, and a number of times with ether, dry at the temperature of boiling water for 3 hours, and weigh. Multiply the weight of the mucic acid by 1.33, which gives galactose, and multiply this product by 0.9 which gives galactan.

CRUDE FIBER.—OFFICIAL.

67

REAGENTS.

- (a) 1.25% sulphuric acid solution.—Exact strength, determined by titration.
- (b) 1.25% sodium hydroxid solution.—Exact strength, determined by titration.

68

DETERMINATION.

Extract a quantity of the substance, representing about 2 grams of the dry material, with ordinary ether, or use the residue from the determination of the ether extract. To this residue in a 500 cc. flask add 200 cc. of boiling 1.25% sulphuric acid; connect the flask with an inverted condenser, the tube of which passes only a short distance beyond the rubber stopper into the flask, or simply cover a tall conical flask, which is well suited for this determination, with a watch glass or short stemmed funnel, boil at once and continue boiling gently for 30 minutes. A blast of air conducted into the flask will serve to reduce the frothing of the liquid. Filter through linen and wash with boiling water until the washings are no longer acid; rinse the substance back into the flask with 200 cc. of boiling, 1.25% solution of sodium hydroxid, free or nearly free from sodium carbonate boil at once, and continue boiling gently for 30 minutes as directed above for the treatment with acid, filter at once rapidly, and wash with boiling water until the washings are neutral. The last filtration may be performed upon a Gooch crucible, a linen filter, or a tared filter paper. If a linen filter is used, rinse the crude fiber, after washing is completed, into a flat-bottomed platinum dish by means of a jet of water; evaporate to dryness on a steam bath, dry to constant weight at 110°C., weigh, incinerate completely, and weigh again. The loss in weight is considered to be crude fiber. If a tared filter

paper is used, weigh in a weighing bottle. In any case the crude fiber after drying to constant weight at 110°C. must be incinerated and the amount of the ash deducted from the original weight.

69

WATER-SOLUBLE ACIDITY OF FEEDS.—TENTATIVE.

Weigh 10 grams of the sample into a shaking bottle, add 200 cc. of water, and shake for 15 minutes. Filter the extract through a folded filter and take a 20 cc. aliquot (equivalent to 1 gram of sample) for the titration. Dilute with 50 cc. of water and titrate with N/10 sodium hydroxid, using phenolphthalein as indicator.

In reporting the acidity of feeds, state the results in terms of cc. of N/10 sodium hydroxid required for neutralisation.

BIBLIOGRAPHY.

¹ Z. Ver. Zucker-Ind., 1900, **37** (I): 357; 1913, **63** (I): 25; J. Ind. Eng. Chem., 1913, **5**: 167.

² J. Am. Chem. Soc., 1914, **36**: 1566.

³ Ibid., 1906, **28**: 663; 1907, **29**: 541.

⁴ Ibid., 1902, **24**: 1082.

⁵ Z. Rubenzucker-Ind., 1885, **35** (N. F. **22**): 1012.

⁶ Ibid., 1889, **39** (N. F. **26**): 734.

⁷ Ibid., 1879, **29** (N. F. **16**): 1034.

⁸ Wein. Tables for the Quantitative Estimation of the Sugars. Translated by Frew. 1896, p. 26.

⁹ Ibid., p. 33.

¹⁰ Z. Rubenzucker-Ind., 1882, **32** (N. F. **19**): 606, 865.

¹¹ U. S. Bur. Chem. Circ. 71.

¹² J. Am. Chem. Soc., 1904, **26**: 266.

¹³ U. S. Bur. Chem. Bull. 73, p. 173.

¹⁴ J. Landw., 1900, **48**: 379.

IX. SACCHARINE PRODUCTS.

1

PREPARATION OF SAMPLE.—TENTATIVE.

(a) *Liquids (molasses, sirups, etc.)*.—Mix materials of this class thoroughly. If crystals of sugar are present, dissolve them either by heating gently or by weighing the whole mass, then adding water, heating until completely dissolved and after cooling, re-weighing. Calculate all results to the weight of the original substance.

(b) *Semisolids (jellies, jams, etc.)*.—Weigh 50 grams of the sample into a 250 cc. graduated flask. Treat with water, fill to the mark and mix thoroughly. If insoluble material remains, mix uniformly by shaking before taking aliquots for the various determinations.

(c) *Solids (sugar, confectionery, etc.)*.—Grind and mix thoroughly materials of this class to secure uniform samples.

MOISTURE.

DRYING METHODS.

2

SUGARS.—OFFICIAL.

Dry 2-5 grams in a flat dish (nickel, platinum, or aluminium) at the temperature of boiling water for 10 hours; cool in a desiccator and weigh; then dry again for an hour or until there is only a slight change in weight.

With some sugars, more especially those of large grain, there is danger of occlusion and retention of water. The International Commission for Unifying Methods of Sugar Analysis prescribe drying at 105°-110°C. for normal beet sugars. This temperature is sufficient to expel the last traces of occluded water and is not attended with sufficient decomposition to affect the weight of the product. The drying temperature should never exceed 110°C¹.

MASSECUITES, MOLASSES, AND OTHER LIQUID AND SEMILIQUID PRODUCTS.

3

Drying upon Pumice Stone.—Tentative.

Prepare pumice stone of two grades of fineness, one of which will pass through a 1 mm. sieve, the other through a 6 mm. sieve. Make the determination in flat metallic dishes or in shallow, flat-bottomed, weighing bottles. Place a layer of the fine pumice stone, 3 mm. in thickness, on the bottom of the dish, then a layer of the coarse pumice stone 6-10 mm. in thickness, dry and weigh. Dilute the sample with a weighed portion of water so that the diluted material shall contain 20-30% of solid matter. Weigh into the dish, prepared as described above, an amount of the diluted sample to yield, approximately, 1 gram of dry matter. If this weighing can not be made rapidly, use a weighing bottle provided with a cork through which a pipette passes. Dry in vacuo at 70°C. to constant weight, making trial weighings at intervals of 2 hours. For substances containing little or no levulose or other readily decomposable substance, the drying may be made in a water oven at the temperature of boiling water.

4

Drying upon Quartz Sand.—Tentative.

Digest pure quartz sand with strong hydrochloric acid, wash, dry, and ignite. Preserve in a stoppered bottle.

Place 6-7 grams of the prepared sand and a short stirring rod in a flat-bottomed dish. Dry thoroughly, cool in a desiccator, and weigh. Then add 3-4 grams of the molasses, mix with the sand (if necessary to thoroughly incorporate the two, add a little water), dry in a water oven at the temperature of boiling water for 8-10 hours, stirring at intervals of an hour, cool in a desiccator, and weigh. Stir, heat again for an hour, cool, and weigh. Repeat the heating and weighing until the loss of water in an hour is not greater than 3 mg.

AREOMETRIC METHODS.

(Not applicable to low-grade sugar products, molasses and other materials containing large amounts of non-sugar solids.)

SPECIFIC GRAVITY, WATER AND TOTAL SOLIDS.

5

By Means of a Spindle.—Official.

The density of juices, sirups, etc., is most conveniently determined by means of the Brix hydrometer. For rough work, or where less accuracy is desired, the Baumé hydrometer may be used. The Brix spindle should be graduated to tenths. The range of degrees recorded by each individual spindle should be as limited as possible. The solution should be as nearly as practicable of the same temperature as the air at the time of reading, and, if the variation from the temperature of the graduation of the spindle amounts to more than 1°, a correction must be applied according to the table under 6. Before taking the density of a juice, allow it to stand in the cylinder until all air bubbles have escaped, and until all fatty or waxy matter has come to the surface and been skimmed off. The cylinder should be large enough in diameter to allow the hydrometer to come to rest without touching the sides. A table of specific gravities at $\frac{20^{\circ}\text{C.}}{4^{\circ}}$ and per cent by weight of sucrose is given under 9, and a table for the comparison of specific gravities at $\frac{17.5^{\circ}\text{C.}}{17.5^{\circ}}$, degrees Brix (per cent by weight of sucrose), and degree Baumé is given under 8.

If the sample is too dense to determine the density directly, dilute a weighed portion with a weighed quantity of water, or dissolve a weighed portion and dilute to a known volume with water.

In the first instance the per cent of total solids is calculated by the following formula:

$$\text{Per cent of solids in the undiluted material} = \frac{WS}{w} \text{ in which}$$

S = per cent of solids in the diluted material;

W = weight of the diluted material;

w = weight of the sample taken for dilution.

When the dilution is made to a definite volume, the following formula is to be used:

$$\text{Per cent of solids in the undiluted material} = \frac{VDS}{W} \text{ in which}$$

V = volume of the diluted solution at a given temperature;

D = specific gravity of the diluted solution at the same temperature;

S = per cent of solids in the diluted solution at the same temperature;

W = weight of the sample taken for dilution at the same temperature.

If the spindle reading be made at any other temperature than 17.5°C., the result should be corrected according to the following:

6

TABLE 9.

For correction of the readings of the Brix spindle when made at other than the standard temperature, 17.5°C.

(For temperatures below 17.5°C. the correction is to be subtracted.)

TEMPERATURE	DEGREES BRIX OF THE SOLUTION												
	0	5	10	15	20	25	30	35	40	50	60	70	75
0	0.17	0.30	0.41	0.52	0.62	0.72	0.82	0.92	0.98	1.11	1.22	1.25	1.29
5	0.22	0.30	0.37	0.44	0.52	0.59	0.65	0.72	0.75	0.80	0.85	0.91	0.94
10	0.20	0.26	0.29	0.33	0.36	0.39	0.42	0.45	0.48	0.50	0.54	0.58	0.61
11	0.18	0.23	0.26	0.28	0.31	0.34	0.36	0.39	0.41	0.43	0.47	0.50	0.53
12	0.16	0.20	0.22	0.24	0.26	0.29	0.31	0.33	0.34	0.36	0.40	0.42	0.46
13	0.14	0.18	0.19	0.21	0.22	0.24	0.26	0.27	0.28	0.29	0.33	0.35	0.39
14	0.12	0.15	0.16	0.17	0.18	0.19	0.21	0.22	0.22	0.23	0.26	0.28	0.32
15	0.09	0.11	0.12	0.14	0.14	0.15	0.16	0.17	0.16	0.17	0.19	0.21	0.25
16	0.06	0.07	0.08	0.09	0.10	0.10	0.11	0.12	0.12	0.12	0.14	0.16	0.18
17	0.02	0.02	0.03	0.03	0.03	0.04	0.04	0.04	0.04	0.04	0.05	0.05	0.06
18	0.02	0.02	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.02
19	0.06	0.08	0.08	0.09	0.09	0.10	0.10	0.10	0.10	0.10	0.10	0.08	0.06
20	0.11	0.14	0.15	0.17	0.17	0.18	0.18	0.18	0.19	0.19	0.18	0.15	0.11
21	0.16	0.20	0.22	0.24	0.24	0.25	0.25	0.25	0.26	0.26	0.25	0.22	0.18
22	0.21	0.26	0.29	0.31	0.31	0.32	0.32	0.32	0.33	0.34	0.32	0.29	0.25
23	0.27	0.32	0.35	0.37	0.38	0.39	0.39	0.39	0.40	0.42	0.39	0.36	0.33
24	0.32	0.38	0.41	0.43	0.44	0.46	0.46	0.47	0.47	0.50	0.46	0.43	0.40
25	0.37	0.44	0.47	0.49	0.51	0.53	0.54	0.55	0.55	0.58	0.54	0.51	0.48
26	0.43	0.50	0.54	0.56	0.58	0.60	0.61	0.62	0.62	0.66	0.62	0.59	0.55
27	0.49	0.57	0.61	0.63	0.65	0.68	0.68	0.69	0.70	0.74	0.70	0.65	0.62
28	0.56	0.64	0.68	0.70	0.72	0.76	0.76	0.78	0.78	0.82	0.78	0.72	0.70
29	0.63	0.71	0.75	0.78	0.79	0.84	0.84	0.86	0.86	0.90	0.86	0.80	0.78
30	0.70	0.78	0.83	0.87	0.87	0.92	0.92	0.94	0.94	0.98	0.94	0.88	0.86
35	1.10	1.17	1.22	1.24	1.30	1.32	1.33	1.35	1.36	1.39	1.34	1.27	1.25
40	1.50	1.61	1.67	1.71	1.73	1.79	1.79	1.80	1.82	1.83	1.78	1.69	1.65
50	2.65	2.71	2.74	2.78	2.80	2.80	2.80	2.80	2.79	2.70	2.56	2.51
60	3.87	3.88	3.88	3.88	3.88	3.88	3.88	3.90	3.82	3.70	3.43	3.41
70	5.17	5.18	5.20	5.14	5.13	5.10	5.08	5.06	4.90	4.72	4.47	4.36
80	6.62	6.59	6.54	6.46	6.38	6.30	6.26	6.06	5.82	5.50	5.33
90	8.26	8.16	8.06	7.97	7.83	7.71	7.58	7.30	6.96	6.58	6.37
100	10.01	9.87	9.72	9.56	9.39	9.21	9.03	8.64	8.22	7.76	7.42

Example.—A sugar solution shows a reading of 30.2° Brix at 30°C. To find the necessary correction for the conversion of this reading to the reading which would have been obtained if the observation had been made at 17.5°C., find the vertical column in the table headed 30° Brix, which is the nearest to the observed reading. Follow down this column until the number is reached which is opposite to the temperature of observation—in this case 30°. The number found, 0.92, is to be added to the observed reading.

7

By Means of a Pycnometer.—Official.

(a) *By specific gravity at $\frac{20^\circ\text{C.}}{4^\circ}$.*—Determine the specific gravity of the solution at $\frac{20^\circ\text{C.}}{4^\circ}$ by means of a pycnometer and ascertain the corresponding per cent by weight of sucrose from 9. When the density of the substance is too high for a direct determination, dilute and calculate the sucrose content of the original material as directed under 5.

(b) *By specific gravity at $\frac{17.5^\circ\text{C.}}{17.5^\circ}$.*—Proceed as directed under (a), the determinations of specific gravity being made at $\frac{17.5^\circ\text{C.}}{17.5^\circ}$ instead of at $\frac{20^\circ\text{C.}}{4^\circ}$. Ascertain the corresponding per cent by weight of sucrose from 8.

The pycnometer determination should not be made at any other temperature than $\frac{17.5^\circ\text{C.}}{17.5^\circ}$ or $\frac{20^\circ\text{C.}}{4^\circ}$.

TABLE 10.

For the comparison of specific gravities at $\frac{17.5^{\circ}\text{C.}}{17.5^{\circ}}$, degrees Brix and degrees Baumé.

$$\text{Degree Baumé} = 146.78 - \frac{146.78}{\text{sp. gr.}}$$

DEGREE BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY	DEGREE BAUMÉ	DEGREE BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY	DEGREE BAUMÉ	DEGREE BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY	DEGREE BAUMÉ
1.0	1.00388	0.6	33.0	1.14423	18.5	65.0	1.31989	35.6
2.0	1.00779	1.1	34.0	1.14915	19.05	66.0	1.32601	36.1
3.0	1.01173	1.7	35.0	1.15411	19.6	67.0	1.33217	36.6
4.0	1.01570	2.3	36.0	1.15911	20.1	68.0	1.33836	37.1
5.0	1.01970	2.8	37.0	1.16413	20.7	69.0	1.34460	37.6
6.0	1.02373	3.4	38.0	1.16920	21.2	70.0	1.35088	38.1
7.0	1.02779	4.0	39.0	1.17430	21.8	71.0	1.35720	38.6
8.0	1.03187	4.5	40.0	1.17943	22.3	72.0	1.36355	39.1
9.0	1.03599	5.1	41.0	1.18460	22.9	73.0	1.36995	39.6
10.0	1.04014	5.7	42.0	1.18981	23.4	74.0	1.37639	40.1
11.0	1.04431	6.2	43.0	1.19505	23.95	75.0	1.38287	40.6
12.0	1.04852	6.8	44.0	1.20033	24.5	76.0	1.38939	41.1
13.0	1.05276	7.4	45.0	1.20565	25.0	77.0	1.39595	41.6
14.0	1.05703	7.9	46.0	1.21100	25.6	78.0	1.40254	42.1
15.0	1.06133	8.5	47.0	1.21639	26.1	79.0	1.40918	42.6
16.0	1.06566	9.0	48.0	1.22182	26.6	80.0	1.41586	43.1
17.0	1.07002	9.6	49.0	1.22728	27.2	81.0	1.42258	43.6
18.0	1.07441	10.1	50.0	1.23278	27.7	82.0	1.42934	44.1
19.0	1.07884	10.7	51.0	1.23832	28.2	83.0	1.43614	44.6
20.0	1.08329	11.3	52.0	1.24390	28.8	84.0	1.44298	45.1
21.0	1.08778	11.8	53.0	1.24951	29.3	85.0	1.44986	45.5
22.0	1.09231	12.4	54.0	1.25517	29.8	86.0	1.45678	46.0
23.0	1.09686	13.0	55.0	1.26086	30.4	87.0	1.46374	46.5
24.0	1.10145	13.5	56.0	1.26658	30.9	88.0	1.47074	47.0
25.0	1.10607	14.1	57.0	1.27235	31.4	89.0	1.47778	47.45
26.0	1.11072	14.6	58.0	1.27816	31.9	90.0	1.48486	47.9
27.0	1.11541	15.2	59.0	1.28400	32.5	91.0	1.49199	48.5
28.0	1.12013	15.7	60.0	1.28989	33.0	92.0	1.49915	48.9
29.0	1.12488	16.3	61.0	1.29581	33.5	93.0	1.50635	49.4
30.0	1.12967	16.8	62.0	1.30177	34.0	94.0	1.51359	49.8
31.0	1.13449	17.4	63.0	1.30777	34.5	95.0	1.52087	50.3
32.0	1.13934	17.95	64.0	1.31381	35.1			

When the number expressing the specific gravity found by analysis falls between the numbers given in the above table, the exact equivalent in degrees Brix or Baumé is found by a simple calculation.

Example.—The pycnometer shows the specific gravity of a certain sirup to be 1.20909. The table shows that the corresponding degree Brix is between 45.0 and 46.0. Subtracting the specific gravity of a solution of 45° Brix from the corresponding figure for 46°, we have (expressing the specific gravities as whole numbers) $121,100 - 120,565 = 535$, the difference in specific gravity for 1° Brix at this point in the table. Subtracting the specific gravity corresponding to 45° from the specific gravity found by analysis, we have $120,909 - 120,565 = 344$; $\frac{344}{535} = 0.64$, the fraction of 1° Brix more than 45°. The degree Brix, corresponding to a sp. gr. of 1.20909, is therefore 45.64.

9

TABLE 11.

Densities² of solutions of cane sugar at 20°C.

(This table is the basis for standardizing hydrometers indicating per cent of sugar at 20°C.)

PER CENT SUGAR	TENTHS OF PER CENT									
	0	1	2	3	4	5	6	7	8	9
0	0.998324	0.998623	0.999010	0.999398	0.999786	1.000174	1.000563	1.000952	1.001343	1.001731
1	1.002120	1.002509	1.002897	1.003286	1.003675	1.004064	1.004453	1.004844	1.005234	1.005624
2	1.006015	1.006405	1.006796	1.007189	1.007580	1.007972	1.008363	1.008755	1.009148	1.009541
3	1.009934	1.010327	1.010721	1.011115	1.011510	1.011904	1.012298	1.012694	1.013089	1.013485
4	1.013881	1.014277	1.014673	1.015070	1.015467	1.015864	1.016261	1.016659	1.017058	1.017456
5	1.017854	1.018253	1.018652	1.019053	1.019451	1.019851	1.020251	1.020651	1.021053	1.021454
6	1.021855	1.022257	1.022659	1.023061	1.023463	1.023867	1.024270	1.024673	1.025077	1.025481
7	1.025885	1.026289	1.026694	1.027099	1.027504	1.027910	1.028316	1.028722	1.029128	1.029535
8	1.029942	1.030349	1.030757	1.031165	1.031573	1.031983	1.032390	1.032800	1.033209	1.033619
9	1.034029	1.034439	1.034850	1.035260	1.035671	1.036082	1.036494	1.036906	1.037318	1.037730
10	1.038143	1.038556	1.038970	1.039383	1.039797	1.040212	1.040626	1.041041	1.041456	1.041873
11	1.042288	1.042704	1.043121	1.043537	1.043954	1.044370	1.044788	1.045206	1.045625	1.046043
12	1.046462	1.046881	1.047300	1.047720	1.048140	1.048559	1.048980	1.049401	1.049823	1.050243
13	1.050665	1.051087	1.051510	1.051933	1.052356	1.052778	1.053202	1.053626	1.054050	1.054475
14	1.054900	1.055325	1.055751	1.056176	1.056602	1.057029	1.057455	1.057882	1.058310	1.058737
15	1.059165	1.059593	1.060022	1.060451	1.060880	1.061306	1.061733	1.062168	1.062608	1.063039
16	1.063460	1.063892	1.064324	1.064756	1.065188	1.065621	1.066054	1.066487	1.066921	1.067355
17	1.067789	1.068223	1.068658	1.069093	1.069529	1.069964	1.070400	1.070836	1.071273	1.071710
18	1.072147	1.072585	1.073023	1.073461	1.073900	1.074338	1.074777	1.075217	1.075657	1.076097
19	1.076537	1.076978	1.077419	1.077860	1.078302	1.078744	1.079187	1.079629	1.080072	1.080516
20	1.080959	1.081403	1.081848	1.082292	1.082737	1.083182	1.083628	1.084074	1.084520	1.084967
21	1.085414	1.085861	1.086309	1.086757	1.087205	1.087653	1.088101	1.088550	1.089000	1.089450
22	1.089900	1.090351	1.090802	1.091253	1.091704	1.092155	1.092607	1.093060	1.093513	1.093966
23	1.094420	1.094874	1.095328	1.095783	1.096236	1.096691	1.097147	1.097603	1.098058	1.098514
24	1.098971	1.099428	1.099886	1.100344	1.100802	1.101259	1.101718	1.102177	1.102637	1.103097
25	1.103557	1.104017	1.104478	1.104938	1.105400	1.105862	1.106324	1.106786	1.107248	1.107711
26	1.108175	1.108639	1.109103	1.109568	1.110033	1.110497	1.110963	1.111429	1.111895	1.112361
27	1.112828	1.113295	1.113763	1.114231	1.114697	1.115166	1.115635	1.116104	1.116573	1.117042
28	1.117512	1.117982	1.118453	1.118923	1.119395	1.119867	1.120339	1.120812	1.121284	1.121757
29	1.122231	1.122703	1.123179	1.123653	1.124128	1.124603	1.125079	1.125555	1.126030	1.126507
30	1.126984	1.127461	1.127939	1.128417	1.128896	1.129374	1.129853	1.130332	1.130812	1.131292
31	1.131773	1.132254	1.132735	1.133216	1.133698	1.134180	1.134663	1.135146	1.135628	1.136111
32	1.136596	1.137080	1.137565	1.138049	1.138534	1.139020	1.139506	1.139993	1.140479	1.140966
33	1.141453	1.141941	1.142429	1.142916	1.143405	1.143894	1.144384	1.144874	1.145363	1.145854
34	1.146345	1.146836	1.147328	1.147820	1.148313	1.148806	1.149298	1.149792	1.150286	1.150780
35	1.151275	1.151770	1.152265	1.152760	1.153256	1.153752	1.154249	1.154746	1.155242	1.155740
36	1.156238	1.156736	1.157235	1.157733	1.158233	1.158733	1.159233	1.159733	1.160233	1.160734
37	1.161236	1.161738	1.162240	1.162742	1.163245	1.163748	1.164252	1.164756	1.165259	1.165764
38	1.166269	1.166775	1.167281	1.167786	1.168293	1.168800	1.169307	1.169815	1.170322	1.170831
39	1.171340	1.171849	1.172359	1.172869	1.173379	1.173889	1.174400	1.174911	1.175423	1.175935
40	1.176447	1.176960	1.177473	1.177987	1.178501	1.179014	1.179527	1.180044	1.180560	1.181076
41	1.181592	1.182108	1.182625	1.183142	1.183660	1.184178	1.184696	1.185215	1.185734	1.186253
42	1.186773	1.187293	1.187814	1.188335	1.188856	1.189379	1.189901	1.190423	1.190946	1.191469
43	1.191993	1.192517	1.193041	1.193565	1.194090	1.194616	1.195141	1.195667	1.196193	1.196720
44	1.197247	1.197775	1.198303	1.198832	1.199360	1.199890	1.200420	1.200950	1.201480	1.202010
45	1.202540	1.203071	1.203603	1.204136	1.204668	1.205200	1.205733	1.206266	1.206801	1.207335
46	1.207870	1.208405	1.208940	1.209477	1.210013	1.210549	1.211086	1.211623	1.212162	1.212700
47	1.213238	1.213777	1.214317	1.214856	1.215395	1.215936	1.216476	1.217017	1.217559	1.218101
48	1.218643	1.219185	1.219729	1.220272	1.220815	1.221360	1.221904	1.222449	1.222995	1.223540
49	1.224086	1.224632	1.225177	1.225727	1.226274	1.226823	1.227371	1.227919	1.228469	1.229018
50	1.229567	1.230117	1.230668	1.231219	1.231770	1.232322	1.232874	1.233426	1.233979	1.234532
51	1.235085	1.235639	1.236194	1.236748	1.237303	1.237859	1.238414	1.238970	1.239527	1.240084
52	1.240641	1.241198	1.241757	1.242315	1.242873	1.243433	1.243992	1.244552	1.245113	1.245673
53	1.246234	1.246795	1.247358	1.247920	1.248482	1.249046	1.249609	1.250172	1.250737	1.251301
54	1.251866	1.252431	1.252997	1.253563	1.254129	1.254697	1.255264	1.255831	1.256400	1.256967
55	1.257535	1.258104	1.258674	1.259244	1.259815	1.260385	1.260955	1.261527	1.262099	1.262671
56	1.263243	1.263816	1.264390	1.264963	1.265537	1.266112	1.266686	1.267261	1.267837	1.268413
57	1.268989	1.269565	1.270143	1.270720	1.271299	1.271877	1.272455	1.273035	1.273614	1.274194
58	1.274774	1.275354	1.275935	1.276517	1.277098	1.277680	1.278262	1.278844	1.279428	1.280011
59	1.280595	1.281179	1.281764	1.282349	1.282935	1.283521	1.284107	1.284694	1.285281	1.285869

TABLE 11.—Continued.

Densities of solutions of cane sugar at 20°C.

PER CENT SUGAR	TENTHS OF PER CENT									
	0	1	2	3	4	5	6	7	8	9
60	1.286456	1.287044	1.287633	1.288222	1.288811	1.289401	1.289991	1.290581	1.291172	1.291763
61	1.292354	1.292946	1.293539	1.294131	1.294725	1.295318	1.295911	1.296506	1.297100	1.297696
62	1.298291	1.298886	1.299483	1.300079	1.300677	1.301274	1.301871	1.302470	1.303068	1.303668
63	1.304267	1.304867	1.305467	1.306068	1.306669	1.307271	1.307872	1.308475	1.309077	1.309680
64	1.310282	1.310885	1.311489	1.312093	1.312699	1.313304	1.313909	1.314515	1.315121	1.315728
65	1.316334	1.316941	1.317549	1.318157	1.318766	1.319374	1.319983	1.320593	1.321203	1.321814
66	1.322425	1.323036	1.323648	1.324259	1.324872	1.325484	1.326097	1.326711	1.327325	1.327940
67	1.328554	1.329170	1.329785	1.330401	1.331017	1.331633	1.332250	1.332868	1.333485	1.334103
68	1.334722	1.335342	1.335961	1.336581	1.337200	1.337821	1.338441	1.339063	1.339684	1.340306
69	1.340928	1.341551	1.342174	1.342798	1.343421	1.344046	1.344671	1.345296	1.345922	1.346547
70	1.347174	1.347801	1.348427	1.349055	1.349682	1.350311	1.350939	1.351568	1.352197	1.352827
71	1.353456	1.354087	1.354717	1.355349	1.355980	1.356612	1.357245	1.357877	1.358511	1.359144
72	1.359778	1.360413	1.361047	1.361682	1.362317	1.362953	1.363590	1.364226	1.364864	1.365501
73	1.366139	1.366777	1.367415	1.368054	1.368693	1.369333	1.369973	1.370613	1.371254	1.371894
74	1.372536	1.373178	1.373820	1.374463	1.375106	1.375749	1.376392	1.377036	1.377680	1.378323
75	1.378971	1.379617	1.380262	1.380909	1.381555	1.382203	1.382851	1.383499	1.384148	1.384796
76	1.385446	1.386096	1.386745	1.387396	1.388045	1.388696	1.389347	1.389997	1.390648	1.391300
77	1.391956	1.392610	1.393263	1.393917	1.394571	1.395226	1.395881	1.396536	1.397192	1.397848
78	1.398505	1.399162	1.399819	1.400477	1.401134	1.401793	1.402453	1.403111	1.403771	1.404430
79	1.405091	1.405753	1.406412	1.407074	1.407735	1.408398	1.409061	1.409723	1.410387	1.411051
80	1.411715	1.412380	1.413044	1.413709	1.414374	1.415040	1.415706	1.416373	1.417039	1.417707
81	1.418374	1.419043	1.419711	1.420380	1.421049	1.421719	1.422390	1.423060	1.423730	1.424400
82	1.425072	1.425744	1.426416	1.427089	1.427761	1.428435	1.429109	1.429782	1.430457	1.431131
83	1.431807	1.432483	1.433158	1.433835	1.434511	1.435188	1.435866	1.436543	1.437222	1.437900
84	1.438579	1.439259	1.439938	1.440619	1.441299	1.441980	1.442661	1.443342	1.444024	1.444705
85	1.445388	1.446071	1.446754	1.447438	1.448121	1.448806	1.449491	1.450175	1.450860	1.451545
86	1.452232	1.452919	1.453605	1.454292	1.454980	1.455668	1.456357	1.457045	1.457735	1.458424
87	1.459114	1.459805	1.460495	1.461186	1.461877	1.462568	1.463260	1.463953	1.464645	1.465338
88	1.466032	1.466726	1.467420	1.468115	1.468810	1.469504	1.470200	1.470896	1.471592	1.472289
89	1.472986	1.473681	1.474381	1.475080	1.475779	1.476477	1.477176	1.477876	1.478575	1.479275
90	1.479976	1.480677	1.481378	1.482080	1.482782	1.483484	1.484187	1.484890	1.485593	1.486297
91	1.487002	1.487707	1.488411	1.489117	1.489823	1.490528	1.491234	1.491941	1.492647	1.493355
92	1.494063	1.494771	1.495479	1.496188	1.496897	1.497606	1.498316	1.499026	1.499736	1.500447
93	1.501158	1.501870	1.502582	1.503293	1.504006	1.504719	1.505432	1.506146	1.506860	1.507574
94	1.508289	1.509004	1.509720	1.510435	1.511151	1.511868	1.512585	1.513302	1.514019	1.514737
95	1.515455	1.516174	1.516893	1.517612	1.518332	1.519051	1.519771	1.520492	1.521212	1.521934
96	1.522656	1.523378	1.524100	1.524823	1.525546	1.526269	1.526993	1.527717	1.528441	1.529166
97	1.529891	1.530616	1.531342	1.532068	1.532794	1.533521	1.534248	1.534976	1.535704	1.536432
98	1.537161	1.537889	1.538618	1.539347	1.540076	1.540806	1.541536	1.542267	1.542998	1.543730
99	1.544462	1.545194	1.545926	1.546659	1.547392	1.548127	1.548861	1.549595	1.550329	1.551064
100	1.551800									

REFRACTOMETER METHOD.—TENTATIVE.

Determine the refractive index of the solution at 28°C. and obtain the corresponding percentage of dry substance from 11. If the refractive index is obtained at a temperature other than 28°C., correct the result as indicated in 12. If the solution is too dark to be read in the instrument, dilute with a concentrated sugar solution. Water should never be used for this purpose. Mix weighed amounts of the solution under examination and a solution of pure sugar of about the same strength, and obtain the amount of dry substance in the former by the following formula:

$$x = \frac{(A + B) C - BD}{A} \text{ in which}$$

x = per cent of dry substance to be found;

A = weight in grams of the material mixed with B ;

B = weight in grams of pure sugar solution employed in the dilution;

C = per cent of dry substance in the mixture of A and B obtained from the refractive index;

D = per cent of dry substance in the pure sugar solution obtained from its refractive index.

11

TABLE 12.—GEERLIGS' TABLE.

For dry substance in sugar-house products by the Abbe refractometer, at 28°C.

INDEX	PER CENT DRY SUB- STANCE	DECIMALS TO BE ADDED FOR FRACTIONAL READINGS*	INDEX	PER CENT DRY SUB- STANCE	DECIMALS TO BE ADDED FOR FRACTIONAL READINGS*	INDEX	PER CENT DRY SUB- STANCE	DECIMALS TO BE ADDED FOR FRACTIONAL READINGS*
1.3335	1	0.0001 = 0.05	1.3484	11	0.0001 = 0.05	1.3746	27	0.0001 = 0.05
1.3349	2	0.0002 = 0.1	1.3500	12	0.0002 = 0.1	1.3764	28	0.0002 = 0.1
1.3364	3	0.0003 = 0.2	1.3516	13	0.0003 = 0.2	1.3782	29	0.0003 = 0.15
1.3379	4	0.0004 = 0.25	1.3530	14	0.0004 = 0.25	1.3800	30	0.0004 = 0.2
1.3394	5	0.0005 = 0.3	1.3546	15	0.0005 = 0.3	1.3818	31	0.0005 = 0.25
1.3409	6	0.0006 = 0.4	1.3562	16	0.0006 = 0.4	1.3836	32	0.0006 = 0.3
1.3424	7	0.0007 = 0.5	1.3578	17	0.0007 = 0.45	1.3854	33	0.0007 = 0.35
1.3439	8	0.0008 = 0.6	1.3594	18	0.0008 = 0.5	1.3872	34	0.0008 = 0.4
1.3454	9	0.0009 = 0.7	1.3611	19	0.0009 = 0.6	1.3890	35	0.0009 = 0.45
1.3469	10	0.0010 = 0.75	1.3627	20	0.0010 = 0.65	1.3909	36	0.0010 = 0.5
		0.0011 = 0.8	1.3644	21	0.0011 = 0.7	1.3928	37	0.0011 = 0.55
		0.0012 = 0.8	1.3661	22	0.0012 = 0.75	1.3947	38	0.0012 = 0.6
		0.0013 = 0.85	1.3678	23	0.0013 = 0.8	1.3966	39	0.0013 = 0.65
		0.0014 = 0.9	1.3695	24	0.0014 = 0.85	1.3984	40	0.0014 = 0.7
		0.0015 = 1.0	1.3712	25	0.0015 = 0.9	1.4003	41	0.0015 = 0.75
			1.3729	26	0.0016 = 0.95			0.0016 = 0.8
								0.0017 = 0.85
								0.0018 = 0.9
								0.0019 = 0.95
								0.0020 = 1.0
								0.0021 = 1.0
1.4023	42	0.0001 = 0.05	1.4292	55	0.0001 = 0.05	1.4711	73	0.0001 = 0.0
1.4043	43	0.0002 = 0.1	1.4314	56	0.0002 = 0.1	1.4736	74	0.0002 = 0.05
1.4063	44	0.0003 = 0.15	1.4337	57	0.0003 = 0.1	1.4761	75	0.0003 = 0.1
1.4083	45	0.0004 = 0.2	1.4359	58	0.0004 = 0.15	1.4786	76	0.0004 = 0.15
1.4104	46	0.0005 = 0.25	1.4382	59	0.0005 = 0.2	1.4811	77	0.0005 = 0.2
1.4124	47	0.0006 = 0.3	1.4405	60	0.0006 = 0.25	1.4836	78	0.0006 = 0.25
1.4145	48	0.0007 = 0.35	1.4428	61	0.0007 = 0.3	1.4862	79	0.0007 = 0.3
1.4166	49	0.0008 = 0.4	1.4451	62	0.0008 = 0.35	1.4888	80	0.0008 = 0.35
1.4186	50	0.0009 = 0.45	1.4474	63	0.0009 = 0.4	1.4914	81	0.0009 = 0.4
1.4207	51	0.0010 = 0.5	1.4497	64	0.0010 = 0.45	1.4940	82	0.0010 = 0.45
1.4228	52	0.0011 = 0.55	1.4520	65	0.0011 = 0.5	1.4966	83	0.0011 = 0.5
1.4249	53	0.0012 = 0.6	1.4543	66	0.0012 = 0.55	1.4992	84	0.0012 = 0.55
1.4270	54	0.0013 = 0.65	1.4567	67	0.0013 = 0.6	1.5019	85	0.0013 = 0.6
		0.0014 = 0.7	1.4591	68	0.0014 = 0.65	1.5046	86	0.0014 = 0.65
		0.0015 = 0.75	1.4615	69	0.0015 = 0.7	1.5073	87	0.0015 = 0.7
		0.0016 = 0.8	1.4639	70	0.0016 = 0.75	1.5100	88	0.0016 = 0.75
		0.0017 = 0.85	1.4663	71	0.0017 = 0.8	1.5127	89	0.0017 = 0.8
		0.0018 = 0.9	1.4687	72	0.0018 = 0.85	1.5155	90	0.0018 = 0.85
		0.0019 = 0.95			0.0019 = 0.9			0.0019 = 0.9
		0.0020 = 1.0			0.0020 = 0.95			0.0020 = 0.95
		0.0021 = 1.0			0.0021 = 1.0			0.0021 = 1.0
					0.0022 = 0.95			0.0022 = 0.95
					0.0023 = 1.0			0.0023 = 1.0
					0.0024 = 1.0			0.0024 = 1.0
								0.0025 = 0.9
								0.0026 = 0.95
								0.0027 = 1.0
								0.0028 = 1.0

* Find in the table the refractive index which is next lower than the reading actually made and note the corresponding whole number for the per cent of dry substance. Subtract the refractive index obtained from the table from the observed reading; the decimal corresponding to this difference, as given in the column so marked, is added to the whole per cent of dry substance as first obtained.

TABLE 13.
Corrections for temperature.

TEMPERATURE OF THE PRISMS IN °C.	DRY SUBSTANCE												
	0	5	10	15	20	25	30	40	50	60	70	80	90
	Subtract—												
20	0.53	0.54	0.55	0.56	0.57	0.58	0.60	0.62	0.64	0.62	0.61	0.60	0.58
21	0.46	0.47	0.48	0.49	0.50	0.51	0.52	0.54	0.56	0.54	0.53	0.52	0.50
22	0.40	0.41	0.42	0.42	0.43	0.44	0.45	0.47	0.48	0.47	0.46	0.45	0.44
23	0.33	0.33	0.34	0.35	0.36	0.37	0.38	0.39	0.40	0.39	0.38	0.38	0.38
24	0.26	0.26	0.27	0.28	0.28	0.29	0.30	0.31	0.32	0.31	0.31	0.30	0.30
25	0.20	0.20	0.21	0.21	0.22	0.22	0.23	0.23	0.24	0.23	0.23	0.23	0.22
26	0.12	0.12	0.13	0.14	0.14	0.15	0.15	0.16	0.16	0.16	0.15	0.15	0.14
27	0.07	0.07	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.07
	Add—												
	0	5	10	15	20	25	30	40	50	60	70	80	90
29	0.07	0.07	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.07
30	0.12	0.12	0.13	0.14	0.14	0.14	0.15	0.15	0.16	0.16	0.16	0.15	0.14
31	0.20	0.20	0.21	0.21	0.22	0.22	0.23	0.23	0.24	0.23	0.23	0.23	0.22
32	0.26	0.26	0.27	0.28	0.28	0.29	0.30	0.31	0.32	0.31	0.31	0.30	0.30
33	0.33	0.33	0.34	0.35	0.36	0.37	0.38	0.39	0.40	0.39	0.38	0.38	0.38
34	0.40	0.41	0.42	0.42	0.43	0.44	0.45	0.47	0.48	0.47	0.46	0.45	0.44
35	0.46	0.47	0.48	0.49	0.50	0.51	0.52	0.54	0.56	0.54	0.53	0.52	0.50

ASH.

13

Method I.—Official.

Heat 5-10 grams of the sample in a 50-100 cc. platinum dish at 100°C. until the water is expelled, add a few drops of pure olive oil, and heat slowly over a flame until swelling ceases. Then place the dish in a muffle and heat at low redness until a white ash is obtained.

14

Method II.—Official.

Carbonize the mass at a low heat, dissolve the soluble salts in hot water, burn the residual mass as directed in 13, add the solution of soluble salts, and evaporate to dryness at 100°C., ignite gently, cool in a desiccator, and weigh.

15

Method III.—Official.

Saturate the sample with sulphuric acid, dry, ignite gently, then burn in a muffle at low redness. Deduct one tenth of the weight of the ash, and calculate the per cent.

16

QUANTITATIVE ANALYSIS OF THE ASH.—OFFICIAL.

Proceed as directed under III.

17

SOLUBLE AND INSOLUBLE ASH.—TENTATIVE.

Ash the material as directed under 13 or 14. Add water to the ash in the platinum dish, heat nearly to boiling, filter through an ashless filter paper, and wash with hot water until the combined filtrate and washings measure about 60 cc. Re-

turn the filter paper and contents to the platinum dish, ignite carefully, and weigh. Calculate the percentages of water-soluble and water-insoluble ash.

18 ALKALINITY OF THE SOLUBLE ASH.—TENTATIVE.

Cool the filtrate from **17** and titrate with N/10 hydrochloric acid, using methyl orange as an indicator.

Express the alkalinity in terms of the number of cc. of N/10 acid per 1 gram of the sample.

19 ALKALINITY OF THE INSOLUBLE ASH.—TENTATIVE.

Add an excess of N/10 hydrochloric acid (usually 10–15 cc.) to the ignited insoluble ash in the platinum dish, under **17**, heat to boiling over an asbestos plate, cool, and titrate the excess of hydrochloric acid with N/10 sodium hydroxid, using methyl orange as an indicator.

Express the alkalinity in terms of the number of cc. of N/10 acid per 1 gram of the sample.

20 MINERAL ADULTERANTS IN THE ASH.—TENTATIVE.

Mix 100 grams of molasses, sirup, honey, or the confectionery solution prepared as directed under **1 (b)** and evaporate to a sirupy consistency, with about 35 grams of concentrated sulphuric acid in a large porcelain evaporating dish. Pass an electric current through it while stirring by placing one platinum electrode in the bottom of the dish near one side and attaching the other to the lower end of the glass rod with which the contents are stirred. Begin with a current of about 1 ampere and gradually increase to 4 (modified from method of Budde and Schou⁴ for determining nitrogen electrolytically). In 10–15 minutes the mass is reduced to a fine dry char, which may be readily burnt to a white ash in the original dish over a free flame or in a muffle.

This method⁴ is preferred to the ordinary method of heating with sulphuric acid, especially in the case of molasses, because, if properly manipulated, it comes quietly into the form of a very finely divided char or powder, especially adapted for subsequent quick ignition.

If an electric current is not available, treat in a large porcelain dish 100 grams of the saccharine solution, evaporated to a sirupy consistency, with sufficient concentrated sulphuric acid to thoroughly carbonize the mass and ignite in the usual manner.

The following adulterants may be present: salts of tin, used in molasses to bleach; mineral pigments, such as chromate of lead in yellow confectionery; oxid of iron, sometimes used to simulate the color of chocolate; and copper. These elements may be detected by the usual qualitative tests.

21 NITROGEN.—TENTATIVE.

Determine nitrogen in 5 grams of the material as directed under **I, 18, 21 or 23**, using a larger quantity of the sulphuric acid if necessary for complete digestion.

SUCROSE.

22 Method I.—Tentative.

(Substances in which the volume of the combined insoluble matter and precipitate from clarifying agents is less than 1 cc. from 26 grams.)

Determine sucrose by polarisation before and after inversion, as directed under **VIII, 14**.

All products which contain dextrose or other reducing sugars in the crystalline form, or in supersaturated solution, exhibit the phenomenon of birotation. The constant rotation only should be employed in the Clerget formula, and to obtain this the solutions prepared for direct polarization should be allowed to stand overnight before making the reading. If it is desired to make the direct reading immediately, the birotation may be destroyed by heating the neutral solution to boiling for a few minutes or by adding a few drops of strong ammonium hydroxid before completing the volume.

23

Method II. (Double dilution method.)—Tentative.

(Substances in which the volume of the combined insoluble matter and precipitate from clarifying agents is more than 1 cc. from 26 grams.)

Weigh out a half normal weight of the sample and make up the solution to 100 cc., employing the appropriate clarifier (basic lead acetate for dark colored confectionery or molasses and alumina cream for light colored confectionery). Also weigh out a normal weight of the sample and make up a second solution with the clarifier to 100 cc. Filter and obtain direct polariscopic readings of both solutions. Invert each solution as directed in 22 and obtain its invert reading.

The true direct polarization of the sample is the product of the two direct readings divided by their difference.

The true invert polarization is the product of the two invert readings divided by their difference.

Calculate the sucrose from the true polarizations thus obtained by the formula given under VIII, 14.

COMMERCIAL GLUCOSE (APPROXIMATE).

24

Method I.—Tentative.

(Substances containing little or no invert sugar.)

Commercial glucose can not be determined accurately owing to the varying amounts of dextrin, maltose, and dextrose present in this product. However, in sirups, in which the amount of invert sugar is so small as not to appreciably affect the result, commercial glucose may be estimated approximately by the following formula:*

$$G = \frac{(a - S) 100}{175} \text{ in which}$$

G = per cent of commercial glucose;

a = direct polarization;

S = per cent of cane sugar.

Express the results in terms of commercial glucose polarizing +175°V.

Method II.—Tentative.

25

(Substances containing invert sugar.†)

Prepare an inverted half normal solution of the substance as directed under VIII, 14 except that after inversion cool the solution, make neutral to phenolphthalein with sodium hydroxid solution, slightly acidify with hydrochloric acid, and treat with 5–10 cc. of alumina cream before making up to the mark. Filter and polarize at 87°C. in a 200 mm. jacketed tube. Multiply the reading by 200 and divide by the factor 163 to express the amount of glucose present in terms of glucose polarizing +175°V.

26

REDUCING SUGARS.—TENTATIVE.

Determine either as dextrose or invert sugar as directed under VIII, 50, 51, 52, 54, or 21, 23, 25, 36 or 39.

27

STARCH.—TENTATIVE.

Measure 25 cc. of a solution or uniform mixture, prepared as directed in 1 (b), (representing 5 grams of the sample) into a 300 cc. beaker, or introduce 5 grams of the finely ground sample (previously extracted with ether if the sample contains much fat) into the beaker, add sufficient water to make the volume 100 cc., heat to about 60°C. (avoiding if possible gelatinizing the starch) and allow to stand for about an hour, stirring frequently to secure complete solution of the sugars. Transfer to a stout wide-mouthed bottle, rinse the beaker with a little warm water, cool, add an equal volume of 95% alcohol, mix, and allow to stand at least an hour. Centrifugalize until the precipitate is closely packed on the bottom of the bottle and decant the supernatant liquid through a hardened filter. Wash the precipitate with successive 50 cc. portions of 50% alcohol by centrifugalizing and decanting through the filter until 3 or 4 drops of the washings give no test for sugar with alphanaphthol as described under 68. Transfer the residue from the bottle and the hardened filter to a large flask and determine starch as directed under VIII, 60.

ETHER EXTRACT IN CONFECTIONERY.

28

Continuous Extraction.—Tentative.

(1) Measure 25 cc. of a 20% mixture or solution, prepared as directed under 1 (b), into a very thin, readily frangible, glass evaporating shell (*Hofmeister Schälchen*), containing 5-7 grams of freshly ignited asbestos fiber; or (2) If impossible to obtain a uniform sample, weigh 5 grams of the mixed finely divided sample into a dish, and wash with water upon the asbestos in the evaporating shell, using, if necessary, a small portion of the asbestos fiber on a stirring rod to transfer the last traces of the sample from the dish to the shell. Dry to constant weight at 100°C., cool, wrap loosely in smooth paper, crush into rather small fragments between the fingers, transfer carefully the crushed mass, exclusive of the paper, to an extraction tube or a fat extraction cartridge. A thin lead disk (bottle cap) may be substituted for the Schälchen. The disk may then be cut into small pieces and placed in the extraction tube. Extract with anhydrous ether or petroleum ether (b. p. 45°-60°C. and without weighable residue) in a continuous extraction apparatus for at least 25 hours. In most cases it is advisable to remove the substance from the extractor after the first 12 hours, grind with sand to a fine powder, and re-extract for the remaining 13 hours. Transfer the extract to a tared flask, evaporate the solvent, dry to constant weight in an oven at 100°C.

29

Roesse-Gottlieb Method.—Tentative.

Substances such as butter-scotch, invariably yield extremely inaccurate results by the above method. In such cases introduce 4 grams of the material, or an amount of a uniform solution equivalent to this amount of the dry substance, into a Röhrig tube or similar apparatus, make up to a volume of 10 cc. with water, add 1.25 cc. of concentrated ammonium hydroxid and mix thoroughly. Add 10 cc. of 95% alcohol and mix. Then add 25 cc. of washed ether and shake vigorously for half a minute; then add 25 cc. of petroleum ether (b. p. below 60°C.), and shake again for half a minute. Allow to stand for 20 minutes or until the separation between the

liquids is complete. Draw off as much as possible of the ether-fat solution (usually 0.5-0.8 cc. will be left) into a weighed flask through a small, rapid filter. The flask should be weighed with a similar one as a counterpoise. Again extract the liquid remaining in the tube, this time with 15 cc. each of ether and petroleum ether, shake vigorously half a minute with each, and allow to settle. Proceed as above, washing the tip of the spigot and the filter with a few cc. of a mixture of equal parts of the 2 ethers (previously mixed and free from deposited water). For absolutely exact results the extraction must be repeated. This third extraction usually yields not more than about 1 mg. of fat, if the previous ether-fat solutions have been drawn off closely, or an amount averaging about 0.02% on a 4 gram charge. Evaporate the ether slowly on a steam bath, then dry the fat in a boiling water oven until the loss in weight ceases. Test the purity of the fat by dissolving in a little petroleum ether. Should a residue remain, wash the fat out completely with petroleum ether, dry the residue, weigh, and deduct the weight.

30**PARAFFIN IN CONFECTIONERY.—TENTATIVE.**

Add to the ether extract in the flask, as above obtained, 10 cc. of 95% alcohol and 2 cc. of sodium hydroxid solution (1 to 1), connect the flask with a reflux condenser, and heat for an hour on the water bath, or until saponification is complete. Remove the condenser and allow the flask to remain on the bath until the alcohol is evaporated and the residue is dry. Dissolve the residue as completely as possible in about 40 cc. of water and heat on the bath, shaking frequently. Wash into a separatory funnel, cool, and extract with 4 successive portions of petroleum ether, which are collected in a tared flask or capsule. Evaporate the petroleum ether and dry in the oven to constant weight.

Any phytosterol or cholesterol present in the fat would be extracted with the paraffin. The amount is so insignificant that it may be disregarded generally. The character of the final residue should, however, be confirmed by determining its melting point, specific gravity, and refractive index.

31 ALCOHOL IN SIRUPS USED IN CONFECTIONERY ("BRANDY DROPS").—TENTATIVE.

Collect in a beaker the sirup from a sufficient number of pieces to yield 30-50 grams of sirup. Strain the sirup into a tared beaker and weigh. Introduce the sirup into a 250-300 cc. distilling flask, dilute with half its volume of water, attach the flask to a vertical condenser and distil almost 50 cc., or as much of the liquid as possible without causing charring. Foaming may be prevented by adding a little tannin, or a piece of paraffin about the size of a pea, to the contents of the distillation flask. Cool the distillate, make up to volume with water, mix well, and ascertain the specific gravity of the liquid by means of a pycnometer, and obtain the corresponding weight of alcohol in the 50 cc. of distillate from XVI, 5. Calculate the per cent by weight of alcohol in the candy filling.

32**COLORING MATTER.—TENTATIVE.**

Proceed as directed under XI.

33**METALS.—TENTATIVE.**

Proceed as directed under XII.

HONEY.*

34

PREPARATION OF SAMPLE.—TENTATIVE.

(a) *Liquid or strained honey*.—If the sample is free from granulation, mix thoroughly by stirring or shaking before drawing weighed portions for the analytical determination. If the honey is granulated, place the container, having the stopper loose, in a water bath, and heat at a temperature not exceeding 50°C. until the sugar crystals dissolve; mix thoroughly, cool, and weigh portions for the analytical determinations. If sediment such as particles of comb, wax, sticks, bees, etc., are present, heat the sample to 40°C. in a water bath and filter through cheese-cloth before weighing portions for analysis.

(b) *Comb honey*.—Cut across the top of the comb, if sealed, and separate completely from the comb by straining through a 40 mesh sieve. When portions of the comb or wax pass through the sieve, heat the sample as in (a) and strain through cloth. If the honey is granulated in the comb, heat until the wax is liquified, stir, cool, remove the wax and take the clear liquid for analysis.

35

MOISTURE.

Weigh 2 grams of the sample into a tared, flat-bottomed aluminium dish, having a diameter of about 60 mm. and containing 10–15 grams of fine quartz sand, which has been previously washed, dried and ignited, and a small glass stirring rod; add 5–10 cc. of water and thoroughly incorporate with the sand and honey mixture by means of the rod; dry the dish and its contents to constant weight in a vacuum oven at a temperature not exceeding 70°C.

36

ASH.—OFFICIAL.

Weigh 5–10 grams of honey into a platinum dish, add a few drops of pure olive oil to prevent spattering, and heat carefully until swelling ceases and then ignite at a temperature not above dull redness until a white ash is obtained.

37

SOLUBLE ASH.—TENTATIVE.

Proceed as directed under 17.

38

ALKALINITY OF THE SOLUBLE ASH.—TENTATIVE.

Proceed as directed under 18.

POLARIZATION.

39

Direct Polarization.—Tentative.

(a) *Immediate direct polarization*.—Transfer 26 grams of the honey to a 100 cc. flask with water, add 5 cc. of alumina cream, dilute to the mark with water at 20°C., filter, and polarize immediately in a 200 mm. tube.

(b) *Constant direct polarization*.—Pour the solution from the tube used in reading (a) back into the flask, stopper, and allow to stand for 24 hours. At the end of this time again polarize the solution at 20°C. in a 200 mm. tube.

(c) *Birotation*.—The difference between (a) and (b) gives the birotation.

(d) *Direct polarization at 87°C.*—Polarize the solution, obtained in (b), at 87°C. in a jacketed 200 mm. tube.

40

Invert Polarization.—Tentative.

(a) *At 20°C.*—Invert 50 cc. of the solution obtained in 39 as directed under VIII, 14 or 16, and polarize at 20°C. in a 200 mm. tube.

(b) *At 87°C.*—Polarize the solution, obtained as directed in (a), at 87°C. in a 200 mm. jacketed tube.

41

REDUCING SUGARS.—TENTATIVE.

Dilute 10 cc. of the solution, used for direct polarization, 39, to 250 cc. and determine reducing sugars in 25 cc. of this solution by one of the methods given under VIII, 25, 36, 39 or 56, respectively. Calculate the result to per cent of invert sugar.

42

SUCROSE.—TENTATIVE.

Proceed as directed under VIII, 18. Determine reducing sugars after inversion by diluting 10 cc. of the solution obtained in 40, with a small amount of water, neutralizing with sodium carbonate, and making up to 250 cc. with water. Employ 50 cc. of this solution for the determination, using the same method as in 41.

43

LEVULOSE.—TENTATIVE.

Multiply the direct reading at 87°C., 39 (d), by 1.0315 and subtract the product from the constant direct polarization at 20°C., 39 (b); divide the difference by 2.3919 to obtain the grams of levulose in a normal weight of the honey. From this figure calculate the per cent of levulose in the original sample.

44

DEXTROSE.—TENTATIVE.

Subtract the per cent of levulose, obtained in 43, from the per cent of invert sugar, found in 41, to obtain the approximate per cent of dextrose.

The dextrose can be determined more accurately by multiplying the per cent of levulose, as found in 43, by the factor 0.915, which gives its dextrose equivalent in copper reducing power. Subtract this figure from that of the reducing sugars, 41, calculated as dextrose, to obtain the percentage of dextrose in the sample. (Owing to the difference in the reducing powers of different sugars, the sum of the dextrose thus found and the levulose as obtained in 43 will be greater than the amount of invert sugar obtained in 41).

45

DEXTRIN (APPROXIMATE).—TENTATIVE.

Transfer 8 grams of the sample (4 grams in the case of dark colored honey-dew honey) to a 100 cc. flask (using not more than 4 cc. of water) by allowing the sample to drain from the weighing dish into the flask and then dissolving the residue in 2 cc. of water. After adding this solution to the contents of the flask, rinse the weighing dish with two 1 cc. portions of water to which a little alcohol is added subsequently. Fill the flask to the mark with absolute alcohol, shaking constantly. Set the flask aside until the dextrin has collected on the sides and bottom and the liquid is clear. Decant the clear liquid through a filter paper and wash the residue in the flask with 10 cc. of 95% alcohol, pouring the washings through the same filter. Dissolve the dextrin in the flask with boiling water and filter through the filter paper already used, receiving the filtrate in a tared dish, prepared as directed under 4. Rinse the flask and wash the filter a number of times with small portions of hot water, evaporate on a water bath and dry to constant weight in vacuo at 70°C.

After determining the weight of the alcohol precipitate, dissolve the latter in water and make up to definite volume, using 50 cc. of water for each 0.5 gram of precipitate or part thereof.

Determine reducing sugars in the solution both before and after inversion as directed under VIII, 18, expressing the results as invert sugar. Calculate sucrose from the results thus obtained and subtract the sum of the reducing sugars before inversion and sucrose from the weight of the total alcoholic precipitate to obtain the weight of the dextrin.

46

FREE ACID.—TENTATIVE.

Dissolve 10 grams of the honey in water and titrate with N/10 sodium hydroxid using phenolphthalein as an indicator. Express the results in terms of cc. of N/10 sodium hydroxid required to neutralize 100 grams of the sample.

47

GLUCOSE.—TENTATIVE.

Qualitative test.—Dilute the honey with water in the proportion of 1 to 1, then add a few cc. of iodine solution (1 gram of iodine, 3 grams of potassium iodide, 50 cc. of water). In the presence of glucose the solution turns red or violet, the depth and character of the color depending upon the quality and nature of the glucose employed. A blank test with a pure honey of about the same color should be made in order to secure an accurate color comparison. Should the honey be dark and the percentage of glucose very small, precipitate the dextrin which may be present by adding several volumes of 95% alcohol. Allow to stand until the precipitate settles (do not filter), decant the liquid, dissolve the residue of dextrins in hot water, cool and apply the above test to this solution. A negative result is not proof of the absence of glucose as some glucose, especially of high conversion, does not give any reaction with iodine.⁹

Quantitative test.—An approximate determination can be made by Browne's formula as follows: Multiply the difference in the polarizations of the invert solution at 20°C. and 87°C. by 77 and divide this product by the percentage of invert sugar after inversion found in the sample. Multiply the quotient by 100 and divide the product by 26.7, to obtain the percentage of honey in the sample; 100 per cent minus the per cent of honey gives the percentage of glucose.

COMMERCIAL INVERT SUGAR.¹⁰

QUALITATIVE TESTS.

Fiehe Test (Bryan Modification¹¹).—Tentative.

48

REAGENT.

Resorcin solution.—Dissolve 1 gram of resorcin in 100 cc. of hydrochloric acid, sp. gr. 1.19.

49

MANIPULATION.

Introduce 10 cc. of a 50% honey solution into a test tube and add 5 cc. of ether. Shake gently and allow to stand for some time until the ether layer is clear. Transfer 2 cc. of this clear ether solution to a small test tube and add a large drop of the resorcin solution. Shake and note the color immediately. In the presence of artificial invert sugar, the resorcin assumes immediately an orange-red color turning to dark red.

*Feder Anilin Chlorid Test.*¹²—*Tentative.*

50

REAGENT.

Anilin chlorid solution.—To 100 cc. of C. P. anilin add 30 cc. of 25% hydrochloric acid.

51

MANIPULATION.

Introduce 5 grams of the honey into a porcelain dish and add 2.5 cc. of the anilin reagent. A bright red color indicates the presence of commercial invert sugar.

52

DIASTASE.¹³

Mix 1 part of honey with 2 parts of sterile water. Treat 10 cc. of this solution with 1 cc. of 1% soluble starch solution and digest at 45°C. for an hour. At the end of this time test the mixture with 1 cc. of iodine solution (1 gram of iodine, 2 grams of potassium iodide, 300 cc. of water). Treat another 10 cc. portion of the honey solution, mixed with 1 cc. of the soluble starch solution, without heating to 45°C., with the reagent and compare the colors produced. If the original honey had not been heated sufficiently to kill the diastase, an olive-green or brown coloration will be produced in the mixture that has been heated at 45°C. Heated or artificial honey becomes blue.

MAPLE PRODUCTS.

53

PREPARATION OF SAMPLE.—TENTATIVE.

(a) *Maple sirup.*—Determine the moisture by the method given under 54 (a). If the moisture is less than 35%, and there is some mineral sediment, pour the clear sirup into a beaker, washing the sediment also into the beaker with water. Then concentrate the sirup by boiling to a moisture content of about 35% (b. p. 104°C.). Set aside until cool, or preferably let the covered material stand overnight, and pour off the clear liquid for the analytical work. Where no sediment is present the sample is ready for analysis after careful mixing. Where sugar has crystallized out, warm to dissolve the sugar before starting the analysis. It is desirable in order to compare results upon different samples, to reduce all results other than moisture to a dry substance basis as determined in the clear sirup.

(b) *Maple sugar, maple cream, maple wax, etc.*—Determine moisture, by the method given under 54 (b), in the sample in its original condition by thoroughly mixing, if semi-plastic, or by rubbing up in a mortar representative portions of the product if solid. For all other analytical determinations use a solution prepared as follows: Weigh roughly 100 grams of the product into a beaker and dissolve by boiling with 200 cc. of water. Decant the resulting sirup while hot through a muslin filter, concentrate by boiling to a moisture content of 35% (b. p. 104°C.), cool, or preferably let the covered material stand overnight, set aside until clear, and use this clear sirup for analysis. It is desirable, in order to compare results upon different samples, that all results except moisture be expressed upon a dry basis.

54

MOISTURE.—TENTATIVE.

(a) *Maple sirup.*—Proceed as directed under 35 or 10.

(b) *Maple sugar, maple cream, etc.*—Proceed as directed under 35.

55

POLARIZATION.—TENTATIVE.

(a) *Direct at 20°C.*—Proceed as directed under VIII, 14.

(b) *Invert at 20°C.*—Proceed as directed under VIII, 14.

(c) *Invert at 87°C.*—Proceed as directed under 25 to detect commercial glucose.

56 REDUCING SUGARS AS INVERT SUGAR.—TENTATIVE.

(a) *Before inversion.*—Proceed as directed under VIII, 25, using an aliquot of the solution used for direct polarisation, 55 (a), and only neutral lead acetate for clarification.

(b) *After inversion.*—Proceed as directed under VIII, 25, using an aliquot of the solution used for the invert polarisation, 55 (b), and only neutral lead acetate for clarification.

SUCROSE.**57 By Polarization.—Tentative.**

Proceed as directed under VIII, 14 or 16.

56 By Reducing Sugars Before and After Inversion.—Tentative.

Proceed as directed under VIII, 18.

59 TOTAL ASH.—TENTATIVE.

Proceed as directed under 13.

60 SOLUBLE AND INSOLUBLE ASH.—TENTATIVE.

Proceed as directed under 17.

61 ALKALINITY OF THE SOLUBLE ASH.—TENTATIVE.

Proceed as directed under 18.

62 ALKALINITY OF THE INSOLUBLE ASH.—TENTATIVE.

Proceed as directed under 19.

LEAD NUMBER (WINTON).—TENTATIVE.**63****REAGENTS.**

Standard basic lead acetate solution.—Boil 430 grams of normal lead acetate and 130 grams of litharge, for 30 minutes, or boil 560 grams of Horne's dry basic lead acetate with 1 liter of water, cool, allow to settle and dilute the supernatant liquid to 1.25 sp. gr. To a measured amount of this solution add 4 volumes of water and filter if not perfectly clear. The solution should be standardized each time a set of determinations is made.

If the directions for preparing the basic lead acetate are not carried out carefully, the use of Horne's dry basic lead acetate is preferable.

64**DETERMINATION OF LEAD IN THE BLANK.**

Transfer 25 cc. of the standard basic lead acetate to a 100 cc. flask, add a few drops of acetic acid, and make up to the mark with water. Shake and determine lead sulphate in 10 cc. of the solution as directed under 65. The use of the acid is imperative in this case to keep the lead in solution, when diluted with water.

65**DETERMINATION.**

Transfer 25 grams of the sample to a 100 cc. flask by means of water. Add 25 cc. of the standard basic lead acetate and shake, fill to the mark, shake, and allow to stand for at least 3 hours before filtering. Pipette 10 cc. of the clear filtrate into a 250 cc. beaker, add 40 cc. of water and 1 cc. of concentrated sulphuric acid, shake and add 100 cc. of 95% alcohol. Allow to stand overnight, filter on a tared Gooch,

wash with 95% alcohol, dry in a water oven, and ignite in a muffle or over a Bunsen burner, applying the heat gradually at first, and avoiding a reducing flame. Cool and weigh. Subtract the weight of lead sulphate so found from the weight of lead sulphate found in the blank, 64, and multiply by the factor 27.325. The use of this factor gives the lead number directly without the various calculations otherwise required.

MALIC-ACID VALUE.

66

Cowles Method.¹⁴—Tentative.

Weigh 6.7 grams of the sample into a 200 cc. beaker, add 5 cc. of water, then 2 cc. of a 10% calcium acetate solution and stir. Add gradually, and with constant stirring, 100 cc. of 95% alcohol, and agitate the solution until the precipitate settles, or let stand, until the supernatant liquid is clear. Filter off the precipitate and wash with 75 cc. of 85% alcohol. Dry the filter paper and ignite in a platinum dish. Add 10 cc. of N/10 hydrochloric acid and warm gently until all the lime dissolves. Cool and titrate back with N/10 sodium hydroxid, using methyl orange as an indicator. The difference in cc. divided by 10 represents the malic acid value of the sample. Previous to use the reagents should be tested by a blank determination and any necessary corrections applied.

67

METALS.—TENTATIVE.

Proceed as directed under XII.

SUGAR HOUSE PRODUCTS.

SUCROSE IN BEETS.

68

Alcohol Extraction Method (Herzfeld Modification¹⁵).—Tentative.

Weigh 26 grams of the beet pulp and transfer to a 100 cc. flask with about 50 cc. of 90% alcohol and 3–5 cc. of basic lead acetate solution. Connect a reflux condenser to the flask and place on a boiling water bath for 10–15 minutes. Then pour the whole into a Soxhlet extractor, washing out the flask with fresh portions of 90% alcohol. Connect the same 100 cc. flask to the extractor, and fit the latter with a return condenser. Add 90% alcohol until the siphon is started and the flask is about three fourths full. Place the flask in a covered water bath kept at a heat that will allow the alcohol to boil freely. Continue the extraction for 1–4 hours, or until a test of the alcohol in the extractor gives no color with alpha-naphthol solution when tested as follows: Introduce into a test tube a few drops of the alcohol coming from the extractor, add 4 or 5 drops of a 20% alcoholic alpha-naphthol solution and 2 cc. of water. Shake well, tip the tube, and allow 2–5 cc. of colorless concentrated sulphuric acid to flow down the side of the tube; then hold the tube upright and, if sucrose is present, a color varying from a faint to a deep violet will be noted at the junction of the two liquids. On shaking, the whole solution becomes a blue violet color. This test is suitable for this work, but it must be remembered that other substances besides sucrose give this color reaction.

Remove the flask, transfer to a 100 cc. graduated flask, cool to the standard temperature, dilute to the mark with 90% alcohol, shake and filter, keeping the funnel covered with a watch glass. Polarize in a 200 mm. tube.

Avoid evaporation and changes of temperature and also use a minimum amount of basic acetate for clarification, 3 cc. rather than 5 cc. By digesting the beet pulp with the alcohol before extraction, the time of extraction is greatly shortened, the pulp becomes thoroughly impregnated with the alcohol, and all the air is removed, resulting in a good extraction of the whole material. If the pulp is fine

and tends to clog the siphon, alcohol-washed cotton may be used as a plug in the extractor before adding the beet pulp, and a fine mesh screen may be placed over the pulp to keep the whole compact in the extractor.

69

Pellet Aqueous Method¹⁶ (Hot Digestion).—Tentative.

Weigh 52 grams of the beet cuttings and transfer them with water to a wide-mouthed flask graduated to a content of 201.2 cc.; add 5–10 cc. of basic lead acetate solution, fill the flask to the mark with hot water, and shake. Immerse the flask in a water bath at 80°C. and rotate at intervals. Add water from time to time so that at the end of the heating (about 30 minutes) the water in the flask is a little above the mark. Remove the flask from the water bath and allow it to cool to standard temperature. Add sufficient concentrated acetic acid to make the solution very slightly acid (generally less than 0.5 cc.) and a few drops of ether to break the foam. Make up to the mark, mix thoroughly, filter, and polarize in a 200 mm. tube.

The fineness of the pulp governs the time of heating. Add enough water at the start and maintain this volume during the extraction, so that not more than 5 cc. of water will be necessary to complete the volume after cooling. The proportion of pulp to water must not be increased beyond the prescribed amount, for when smaller proportions of water to pulp are used and then a large quantity of water is added at the last to make up to volume, the sugar does not become equally diffused and the results are too low. Differences of over 1% in sugar content may be caused by lack of care in this particular.

70

Hot Water Digestion Method.—Tentative.

(Hersfeld Modification of the Sachs Le Docte Method¹⁷.)

There are needed nickel-plated sheet iron vessels, 11 cm. high, 6 cm. body diameter, and 4 cm. mouth diameter, also stoppers covered with tin foil to fit the same.

Weigh 26 grams of the beet pulp on a watch glass (small enough to go into the neck of the beaker) and transfer to the metal beaker, add 177 cc. of dilute basic lead acetate solution (5 parts of basic lead acetate solution (sp. gr. 1.25) to 100 parts of water), shake and stopper lightly. Submerge the beaker in a water bath at 75–80°C. for 30 minutes, shaking intermittently. When all the air has been expelled (generally after 5 minutes), tighten the stopper. After 30 minutes, shake, cool to standard temperature, filter, add a drop of acetic acid to the filtrate and polarize in a 400 mm. tube. The reading is the per cent of sugar in the beet pulp.

BIBLIOGRAPHY.

- ¹ Browne. Handbook of Sugar Analysis. 1912, p. 16.
- ² Wiss. Abh. der Kaiserlichen Normal-Eichungs-Kommission, 1900, 2: 153; U. S. Bur. Standards, Circ. 19, 5th ed., p. 26.
- ³ Intern. Sugar J., 10: 69; U. S. Bur. Chem. Bull. 122, p. 169.
- ⁴ Z. anal. Chem., 1899, 38: 345.
- ⁵ Leach. Food Inspection and Analysis. 1913, p. 624.
- ⁶ Analyst, 1896, 21: 182.
- ⁷ Leach. Food Inspection and Analysis. 1913, p. 622.
- ⁸ U. S. Bur. Chem. Bulls. 110 and 154; Z. Nahr. Genussm., 1909, 18: 625.
- ⁹ U. S. Bur. Chem. Bull. 110, p. 60.
- ¹⁰ Ibid., 110 and 154.
- ¹¹ Ibid., 154, p. 15.
- ¹² Analyst, 1911, 36: 586.
- ¹³ Z. Nahr. Genussm., 1910, 19: 72.
- ¹⁴ J. Am. Chem. Soc., 1908, 30: 1285.
- ¹⁵ U. S. Bur. Chem. Bull. 146, p. 17.
- ¹⁶ Ibid., p. 18.
- ¹⁷ Ibid., p. 19.

X. FOOD PRESERVATIVES.—TENTATIVE.

SALICYLIC ACID.

1

PREPARATION OF SAMPLE.

(a) *Non-alcoholic liquids*.—Many liquids may be extracted directly as described in 2 or 4 without further treatment. If gums or mucilaginous substances are present, pipette 100 cc. into a 250 cc. volumetric flask, add about 5 grams of sodium chlorid, shake until the latter is dissolved, make up to the mark with alcohol, shake vigorously, allow the mixture to stand for 10 minutes with occasional shaking, filter through a dry folded filter and treat an aliquot of the filtrate as directed under (b).

(b) *Alcoholic liquids*.—Make 200 cc. of the sample alkaline with sodium hydroxid solution, using litmus as an indicator, and evaporate on a steam bath to about one third its original volume. Dilute to the original volume with water and filter, if necessary, through a dry filter.

(c) *Solid or semi-solid substances*.—Grind the sample and mix thoroughly. Transfer a convenient quantity (50–200 grams according to the consistency of the sample) to a 500 cc. volumetric flask, add sufficient water to make a volume of about 400 cc., shake until the mixture becomes uniform, add 2–5 grams of calcium chlorid, shake until the latter is dissolved, render distinctly alkaline with sodium hydroxid solution, using litmus as an indicator, fill to the mark with water, shake thoroughly, allow to stand for at least 2 hours shaking frequently and filter through a large folded filter.

DETECTION AND ESTIMATION.

2

Ferric Chlorid Test.—Qualitative.

Introduce 50 cc. of the sample or an equivalent amount of an aqueous extract, prepared as directed under 1, into a separatory funnel, add one tenth its volume of dilute hydrochloric acid (1 to 3) and extract with 50 cc. of ether. If the mixture emulsifies, add 10–15 cc. of petroleum ether (b. p. below 60°C.) and shake. If this treatment fails to break the emulsion whirl the mixture in a centrifuge, or allow it to stand until a considerable portion of the aqueous layer has separated, run off the latter, shake vigorously and again allow to separate. Wash the ether layer with two 5 cc. portions of water, evaporate the greater portion of the ether in a porcelain dish on a steam bath, allow the remainder to evaporate spontaneously and add a drop of 0.5% ferric chlorid solution. A violet color indicates salicylic acid.

If coloring matter or other interfering substances are present in the residue left after evaporation of the ether, purify the salicylic acid by one of the following methods:

(a) Dissolve the residue from the ether extract, obtained as directed above, in about 25 cc. of ether, transfer the latter to a separatory funnel and shake with an equal quantity of water, made distinctly alkaline with several drops of ammonium hydroxid. Allow to separate, filter the aqueous layer through a wet filter into a porcelain dish, evaporate almost to dryness, and test the residue as directed above.

(b) Dry the residue from the ether extract, obtained as directed above, in a desiccator over sulphuric acid and extract with several 10 cc. portions of carbon

disulphid or petroleum ether (b. p. below 60°C.), rubbing the contents of the dish with a glass rod, and filtering the successive portions of the solvent through a dry paper into a second porcelain dish. Evaporate the greater portion of the solvent on a steam bath, allow the remainder to evaporate spontaneously and test the residue as directed above.

(C) Transfer the residue from the ether extract, obtained as directed above, to a small porcelain crucible by means of a few cc. of ether and allow the solvent to evaporate spontaneously. Cut a hole in a piece of asbestos board sufficiently large to admit about two thirds of the crucible, cover the latter with a small, round-bottomed flask filled with cold water, and heat over a small Bunsen flame until any salicylic acid present has sublimed and condensed upon the bottom of the flask. Test the sublimate as directed above.

3*Jorissen's Test.*—*Qualitative.*

Dissolve the residue from the ether extract, obtained as directed under 2, or, in case impurities are present, the purified material obtained as directed under 2 (a), (b) or (c) in a little hot water. Cool 10 cc. of the solution in a test tube, add 4 or 5 drops of 10% potassium nitrite solution, 4 or 5 drops of 50% acetic acid and 1 drop of 10% cupric sulphate solution, mix thoroughly and heat to boiling. Boil for half a minute and allow to stand for 1–2 minutes. In the presence of salicylic acid a blood red color will develop.

Colorimetric Method.—*Quantitative.***4****EXTRACTION.**

Pipette a convenient portion of the sample (100 cc. or a volume representing not less than 20 grams of the original sample) or a solution, prepared as in 1, into a separatory funnel, make the solution neutral to litmus with dilute hydrochloric acid (1 to 3) and add an excess of concentrated hydrochloric acid equivalent to 2 cc. of acid for each 100 cc. of solution. Extract with 4 separate portions of ether, using for each extraction a volume of ether equivalent to half the volume of the aqueous layer. If an emulsion forms on shaking, this may usually be broken by adding a little (one fifth the volume of the ether layer) petroleum ether (b. p. below 60°C.) and shaking again or by centrifugalizing. If an emulsion still persists, allow it to remain with the aqueous layer. If an emulsion remains after the fourth extraction, separate it from the clear ether and the clear aqueous layer and extract it separately with 2–3 small portions of ether. Combine the ether extracts, wash with one tenth their volume of water, allow the layers to separate and reject the aqueous layer. Wash in this way until the aqueous layer after separation yields a yellow color upon the addition of methyl orange and 2 drops of N/10 sodium hydroxid. Distil slowly the greater part of the ether, transfer the remainder to a porcelain dish and allow the ether to evaporate spontaneously. If there are no interfering substances present, proceed as directed in 5. If such interfering substances are present, purify the residue by one of the following methods:

(a) Dry thoroughly the residue in vacuo over sulphuric acid and extract with 10 portions of 10–15 cc. each of carbon disulphid or petroleum ether (b. p. below 60°C.), rub the contents of the dish with a glass rod and filter the successive portions of the solvent through a dry filter into a porcelain dish. Test the extracted residue with a drop of ferric alum solution and, if it gives a reaction for salicylic acid, dissolve it in water and reextract with ether, proceeding as directed above. Distil the greater portion of the carbon disulphid or petroleum ether and allow the remainder to evaporate spontaneously. Proceed as directed in 5.

(b) Dissolve the residue in 40-50 cc. of ether. Transfer the ether solution to a separatory funnel and extract with 3 successive 15 cc. portions of 1% ammonium hydroxid. (If fat is known to be present in the original ether extract, extract the latter directly with 4 portions of the ammonium hydroxid instead of 3.) Combine the alkaline aqueous extracts, acidify, again extract with ether and wash the combined ether extracts as directed above. Distil slowly the greater portion of the ether, allow the remainder to evaporate spontaneously and proceed as directed in 5.

5**DETERMINATION.**

Dissolve the residue, obtained in 4, in a small amount of hot water and, after cooling, dilute to a definite volume (usually 50-100 cc.), dependent on the amount of salicylic acid present. If the solution is not clear, filter through a dry filter. Dilute aliquots of the solution and treat with a few drops of 0.5% ferric chlorid solution or 2% ferric alum solution.

The ferric alum solution should be boiled until a precipitate appears, allowed to settle, and filtered. The acidity of the solution is slightly increased in this manner, but it remains clear for a considerable time, and the turbidity caused by its dilution with water is much less and does not appear as soon as when the unboiled solution is used. This turbidity interferes with the exact matching of the color.

Compare the colors developed with that obtained when a standard salicylic acid solution (containing 1 mg. of salicylic acid in 50 cc.) is similarly treated, using Nessler tubes or a colorimeter. In either case, and especially with ferric chlorid, avoid an excess of the reagent, although an excess of 0.5 cc. of 2% ferric alum solution may be added to 50 cc. of the comparison solution of salicylic acid without impairing the results.

BENZOIC ACID.**PREPARATION OF SAMPLE.****6***General Method.*

If solid or semi-solid, grind the sample, and mix thoroughly. Transfer about 150 grams to a 500 cc. graduated flask, add enough pulverized sodium chlorid to saturate the water in the sample, render alkaline with sodium hydroxid solution or milk of lime, and dilute to the mark with a saturated salt solution. Allow to stand for at least 2 hours, with frequent shaking, and filter. If the sample contains large amounts of matter precipitable by salt solution, it is advisable to follow a method similar to that given under 7 (d). When alcohol is present, follow the method given under 7 (c). When large amounts of fats are present, make an alkaline extraction of the filtrate before proceeding as directed under 11.

7*Special Methods.*

(a) *Ketchup*.—Saturate the water in 150 grams of ketchup by adding 15 grams of pulverized sodium chlorid. Transfer the mixture to a 500 cc. graduated flask, rinsing with about 150 cc. of saturated sodium chlorid solution. Make slightly alkaline to litmus paper with strong sodium hydroxid solution and fill to the mark with saturated salt solution. Allow to stand for at least 2 hours, shaking frequently. Squeeze through a heavy muslin bag and then filter through a large folded filter.

(b) *Jellies, jams, preserves and marmalades*.—Dissolve 150 grams of the sample in about 300 cc. of saturated salt solution. Add 15 grams of pulverized sodium chlorid. Make alkaline to litmus paper with milk of lime. Transfer to a 500 cc. graduated flask and dilute to the mark with saturated salt solution. Allow to

stand for at least 2 hours, shaking frequently, centrifugalize if necessary, and filter through a large folded filter.

(C) *Cider containing alcohol, and similar products.*—Make 250 cc. of the sample alkaline to litmus paper with sodium hydroxid solution and evaporate on the steam bath to about 100 cc. Transfer the sample to a 250 cc. graduated flask, add 30 grams of pulverized sodium chlorid and shake until dissolved. Dilute to the original volume, 250 cc., with saturated salt solution, allow to stand for at least 2 hours, shaking frequently, and filter through a folded filter.

(d) *Salted or dried fish.*—Wash 50 grams of the ground sample into a 500 cc. graduated flask with water. Make slightly alkaline to litmus paper with strong sodium hydroxid solution and dilute to the mark with water. Allow to stand for at least 2 hours, shaking frequently, and then filter through a folded filter. Pipette accurately as large a portion of the filtrate as possible (at least 300 cc.) into a second 500 cc. flask. Add 30 grams of the pulverized sodium chlorid for each 100 cc. of solution. Shake until the salt has dissolved and dilute to the mark with saturated salt solution. Mix thoroughly and filter off the precipitated protein matter on a folded filter.

8

DETECTION AND ESTIMATION.

Extract benzoic acid as directed under 2 or 4. If benzoic acid is present in considerable quantity, it will crystallize from the ether in shining leaflets having a characteristic odor on heating. Dissolve the residue in hot water, divide into 2 portions, and test according to 9 or 10.

9

Ferric Chlorid Test.—Qualitative.

Make the solution from 8 alkaline with ammonium hydroxid, expel the excess of ammonia by evaporation, dissolve the residue in water, and add a few drops of a neutral 0.5% ferric chlorid solution. A brownish precipitate of ferric benzoate indicates the presence of benzoic acid.

10

Modified Mohler Test.²—Qualitative.

Add to the water solution, prepared as described under 8, 1–3 cc. of N/3 sodium hydroxid and evaporate to dryness. To the residue, add 5–10 drops of concentrated sulphuric acid and a small crystal of potassium nitrate. Heat for 10 minutes in a glycerol bath at 120°–130°C., or for 20 minutes in a boiling water bath. The temperature must not exceed 130°C. After cooling add 1 cc. of water and make distinctly ammoniacal; boil the solution to decompose any ammonium nitrite which may have been formed. Cool and add a drop of fresh, colorless ammonium sulphid, without allowing the layers to mix. A red-brown ring indicates benzoic acid. On mixing, the color diffuses through the whole liquid and, on heating, finally changes to greenish yellow. This differentiates benzoic acid from salicylic acid or cinnamic acid. The last two form colored compounds, which are not destroyed by heating. The presence of phenolphthalein interferes with this test.

11

Quantitative Method.

Pipette a convenient portion (100–200 cc.) of the filtrate, obtained in 6 or 7, into a separatory funnel. Neutralize the solution to litmus paper with hydrochloric acid (1 to 3) and add an excess of 5 cc. of the same acid. In the case of salted fish a precipitation of protein matter usually occurs on acidifying, but the precipitate does not interfere with the extraction. Extract carefully with chloroform, using successive portions of 70, 50, 40, and 30 cc. To avoid an emulsion, shake cautiously

each time. The chloroform layer usually separates readily after standing a few minutes. If an emulsion forms, break it: (1) by stirring the chloroform layer with a glass rod; (2) by drawing it off into a second funnel and giving 1 or 2 sharp shakes from one end of the funnel to the other; or (3) by centrifugalizing for a few moments. As this is a progressive extraction, draw off carefully as much of the clear chloroform solution as possible after each extraction, but do not draw off any of the emulsion with the chloroform layer. If this precaution is taken, the chloroform extract need not be washed.

Transfer the combined chloroform extracts to a porcelain evaporating dish, rinse the container several times with a few cc. of chloroform, and evaporate to dryness at room temperature in a current of air dried over calcium chlorid.

The extract may also be transferred from the separatory funnel to a 300 cc. Erlenmeyer flask, rinsing the separatory funnel 3 times with 5-10 cc. of chloroform. Distil very carefully to about one fourth the original volume, keeping the temperature down so that the chloroform comes over in drops, not in a steady stream. Then transfer the residue to a porcelain evaporating dish, rinsing the flask 3 times with 5-10 cc. portions of chloroform, and allow to evaporate to dryness spontaneously.

Dry the residue overnight (or until no odor of acetic acid can be detected if the product is a ketchup) in a desiccator containing sulphuric acid. Dissolve the residue of benzoic acid in 30-50 cc. of neutral alcohol, add about one fourth this volume of water, 1 or 2 drops of phenolphthalein, and titrate with N/20 sodium hydroxid (1 cc. is equivalent to 0.0072 gram of anhydrous sodium benzoate).

SACCHARIN.

12

Qualitative Test.

Extract with ether (after maceration and exhaustion with water, if necessary), as directed in 1 and 4. Allow the ether extract to evaporate spontaneously and note the taste of the residue. The presence of saccharin, to the extent of 20 mg. per liter, is indicated by a sweet taste. Confirm by heating with sodium hydroxid, as described below, and detecting the salicylic acid formed thereby. A sweet taste, suggesting the presence of a trace of saccharin, has been obtained frequently in saccharin-free wines, due to the so-called "false saccharin".

Acidify 50 cc. of a liquid food or the aqueous extract of 50 grams of a solid or semi-solid, prepared as directed in 1 (c), and extract with ether as directed in 13. Dissolve the residue, remaining after evaporation of the ether, in a little hot water and test a small portion of this solution for salicylic acid as directed under 2 or 3. Dilute the remainder of the solution to about 10 cc., and add 2 cc. of sulphuric acid (1 to 3). Heat to boiling and add a slight excess of 5% potassium permanganate solution, drop by drop; partly cool the solution, dissolve a piece of sodium hydroxid in it, and filter the mixture into a silver dish (silver crucible lids are well adapted to the purpose); evaporate to dryness and heat for 20 minutes at 210°-215°C. Dissolve the residue in water, acidify with hydrochloric acid and test the ether extract for salicylic acid as directed under 2 or 3. By this method all the so-called "false saccharin" and the salicylic acid naturally present (also added salicylic acid when not present in too large an amount) are destroyed, while 5 mg. of saccharin per liter are detected with certainty.

13

Quantitative Method.

Pipette 100 cc. of the sample, or a convenient portion of a solution, prepared as directed under 1, representing not less than 20 grams of the sample, into a sepa-

ratory funnel, make the solution neutral to litmus with dilute hydrochloric acid (1 to 3) and then add concentrated hydrochloric acid in the proportion of 5 cc. for each 100 cc. of solution. Extract with 4 separate portions of ether using, for each extraction, a volume of ether equivalent to half the volume of the aqueous layer. If the mixture emulsifies on shaking, this difficulty may be overcome as directed under 4. Wash the combined ether extracts with two 5 cc. portions of water, remove the ether by distillation, and transfer the residue into a platinum crucible by means of a small amount of ether. Evaporate the ether on a steam bath, add about 2-3 cc. of 10% sodium carbonate solution to the residue, rotate so that all of the residue is brought into contact with the solution, and evaporate to dryness on a steam bath. Add 4 grams of a mixture of equal parts of anhydrous sodium and potassium carbonates, heat gently at first, and then to complete fusion for 30 minutes over an alcohol or other sulphur-free flame. Cool, dissolve the melt in water, acidify with hydrochloric acid and determine the sulphate present as barium sulphate. Correct the result thus obtained for any sulphur present in the fusion mixture as found in a blank determination. Calculate the amount of saccharin in the sample by multiplying the weight of barium sulphate by 0.7845.

BORIC ACID AND BORATES.

14

Qualitative Test.³

Preliminary test.—Immerse a strip of turmeric paper in the sample acidified with hydrochloric acid in the proportion of 7 cc. of concentrated acid to each 100 cc. of sample, and allow the paper to dry spontaneously. If borax or boric acid is present, the paper will acquire a peculiar red color, changed by ammonium hydroxid to a dark blue-green but restored by acid. Solid or pasty samples may be heated with enough water to make them sufficiently fluid, concentrated hydrochloric acid added in about the proportion of 1 to 13 and the liquid tested in the same way.

Confirmatory test.—Make about 25 grams of the sample decidedly alkaline with lime water and evaporate to dryness on a water bath. Ignite the residue to destroy organic matter. Digest with about 15 cc. of water, add concentrated hydrochloric acid, drop by drop, until the ignited residue is dissolved, and then add 1 cc. in excess. Saturate a piece of turmeric paper with the solution, and allow it to dry without the aid of heat. In the presence of borax or boric acid, the color change will be the same as given above.

15

Quantitative Method.⁴

Make 10-100 grams of the sample (depending upon the nature of the sample and the amount of boric acid present) distinctly alkaline with sodium hydroxid solution and evaporate to dryness in a platinum dish. Ignite the residue until organic matter is destroyed, avoiding an intense red heat, cool, digest with about 20 cc. of hot water, and add hydrochloric acid, drop by drop, until the reaction is distinctly acid. Filter into a 100 cc. flask, and wash with a little hot water, the volume of the filtrate not to exceed 50-60 cc. Return the filter containing any unburned carbon to the platinum dish, make alkaline by wetting thoroughly with lime water, dry on a steam bath and ignite to a white ash. Dissolve the ash in a few cc. of dilute hydrochloric acid and add to the liquid in the 100 cc. flask, rinsing the dish with a few cc. of water. To the combined solutions, add 0.5 gram of calcium chlorid and a few drops of phenolphthalein, then 10% sodium hydroxid solution until a permanent light pink color is produced, and finally dilute to

the mark with lime water. Mix and filter through a dry filter. To 50 cc. of the filtrate add N/1 sulphuric acid until the pink color disappears, then add methyl orange, and continue the addition of the acid until the yellow color is changed to pink. Boil for about 1 minute to expel carbon dioxide. Cool, and carefully add N/5 sodium hydroxid until the liquid assumes a yellow tinge, avoiding an excess of the alkali. All the boric acid is now in a free state with no uncombined sulphuric acid present. Add a little phenolphthalein, and an equal volume of neutral glycerol. Titrate with N/5 sodium hydroxid until a permanent pink color is produced. About 10 grams of mannitol may be substituted for the glycerol in this determination. At the end of the titration add an additional 2 grams and continue the titration if the pink color is discharged. Repeat the alternate addition of mannitol and alkali until a permanent end point is reached.

One cc. of N/5 sodium hydroxid is equivalent to 0.0124 gram of boric acid.

FORMALDEHYDE.

16

PREPARATION OF SAMPLE.

If solid or semi-solid, macerate 200-300 grams of the material with about 100 cc. of water in a mortar. Transfer to a short-necked, 500-800 cc. copper or glass distillation flask and make distinctly acid with phosphoric acid, connect with a condenser and distil 40-50 cc. In the case of highly colored liquids, the same method of preparation should be employed.

In the case of meats and fats, extract the formaldehyde with alcohol and use the filtrate. In the case of fat, heat the mixture above the melting point of the fat to insure thorough extraction. In the case of milk, shake with an equal volume of strong alcohol and use the filtrate. Shake other liquids with an equal volume of strong alcohol and filter from any insoluble matter.

QUALITATIVE TESTS.

17

Phenylhydrazin Hydrochlorid Method.^a

Mix 5 cc. of the distillate, as prepared under 16, or of an alcoholic solution or extract obtained as directed above, with 0.03 gram of phenylhydrazin hydrochlorid, and 4 or 5 drops of a 1% ferric chlorid solution. Add slowly and with agitation, in a bath of cold water to prevent heating the liquid, 1-2 cc. of concentrated sulphuric acid. Dissolve the precipitate by the addition either of concentrated sulphuric acid (keeping the mixture cool) or alcohol. In the presence of formaldehyde a red color develops.

This method gives reliable reactions for formaldehyde in solutions of formaldehyde varying from 1 part in 50,000 to 1 part in 150,000. Acetaldehyde and benzaldehyde give no reaction when treated by this method and do not interfere with the reaction given by formaldehyde.

18

Hehner Method.^a

Mix about 5 cc. of the distillate, obtained in 16, with an equal volume of pure milk, or a 1-2% solution of egg albumen, in a test tube and underlay with strong commercial sulphuric acid without mixing. A violet or blue color at the junction of the two liquids indicates formaldehyde. This color is given only in the presence of a trace of ferric chlorid or other oxidising agent. As pointed out by Hehner, milk may be treated directly by this method and gives positive tests in the presence of 1 or more parts of formaldehyde per 10,000. Some other articles of food rich in proteins, for example, egg albumen, give the reaction in the presence of water without the addition of milk.

19

Leach Method.

Mix about 5 cc. of the distillate, obtained under **16**, with an equal volume of pure milk in a porcelain casserole and add about 10 cc. of concentrated hydrochloric acid, containing 1 cc. of 10% ferric chlorid solution, to each 500 cc. of acid. Heat to 80°-90°C. directly over the gas flame, rotating the casserole to break up the curd. A violet coloration indicates formaldehyde.

Rimini Method.¹

20

Phenylhydrazin Hydrochlorid and Sodium Nitro-prussid Test.

This method may be applied directly to liquid foods, to an aqueous or alcoholic extract of solid foods, or to the distillate prepared as directed in **16**. In the case of milk, apply the method directly. In the case of meat, comminute the sample, extract with 2 volumes of hot water, and employ the expressed liquid for the test. Heat fats above their melting point with 10 cc. of alcohol, shake thoroughly, cool, filter through a moistened filter, and use the filtrate for the test.

Dissolve a lump of phenylhydrazin hydrochlorid about the size of a pea in 3-5 cc. of the liquid to be tested, add 2-4 drops (not more) of a 5-10% sodium nitro-prussid solution and 8-12 drops of an approximately 12% sodium hydroxid solution. If formaldehyde is present, a green or blue color develops depending upon the amount. When formaldehyde is present to the extent of more than 1 part in 70,000-80,000 in the solution tested, a distinct green or bluish green reaction is obtained. In more dilute solutions the green tint becomes less marked and a yellow tinge tending toward greenish brown develops.

With this method acetaldehyde and benzaldehyde give a color varying from red to brown, according to the strength of the solution. A reaction may therefore be obtained with these aldehydes similar to that obtained with formaldehyde in solutions more dilute than 1 part in 70,000. The presence of acetaldehyde or benzaldehyde together with formaldehyde gives a yellowish or yellowish green tinge. The reaction for formaldehyde may therefore be masked by the presence of other aldehydes, but is characteristic when a clear green color is obtained.

21

Phenylhydrazin Hydrochlorid and Potassium Ferricyanid Test.

Proceed as directed in **20**, substituting a solution of potassium ferricyanid for the sodium nitro-prussid. Formaldehyde gives a red color. Alcoholic extracts from foods must be diluted with water to prevent the precipitation of potassium ferricyanid. The test is not applicable in the presence of the coloring matter of blood.

22

Phenylhydrazin Hydrochlorid and Ferric Chlorid Test.

Treat 15 cc. of milk or other liquid food or of the distillate, prepared as directed under **16**, with 1 cc. of a dilute phenylhydrazin hydrochlorid solution, then with a few drops of dilute ferric chlorid solution and, finally, with concentrated hydrochloric acid. The presence of formaldehyde is indicated by the formation of a red color, which changes after some time to orange yellow.

Milk may be examined directly by this method, but more delicate tests may be obtained from the distillate from milk or from milk serum. Acetaldehyde or benzaldehyde does not interfere with the reaction.

23

*Phloroglucol Method.**

To 10 cc. of milk or other liquid food under examination in a test tube add, by means of a pipette, 2 cc. of phloroglucol reagent (1 gram of phloroglucol, 20 grams of sodium hydroxid and water to make 100 cc.), placing the end of the pipette on the bottom of the tube in such a manner that the reagent will form a separate layer.

If formaldehyde be present, a bright red coloration (not purple) forms at the zone of contact. This solution gives a yellow color in the presence of some aldehydes, and, if it is used for the detection of aldehyde formed by the oxidation of methyl alcohol after the destruction of ethyl aldehyde with hydrogen peroxid, an orange yellow color will slowly appear when an insufficient amount of hydrogen peroxid has been employed. On the other hand, if the excess of hydrogen peroxid is not fully destroyed before the use of this reagent, a purple color develops slowly. The clear, red color given by the use of this reagent forms quickly, and, in the presence of but a small amount of formaldehyde fades rapidly.

FLUORIDS.

QUALITATIVE TESTS.

24

Method I.—Modified Method of Blarez.†

Thoroughly mix the sample and boil 150 cc. (in the case of solid foods an aqueous extract may be employed provided the fluorids are in a soluble form). Add to the boiling liquid 5 cc. of 10% potassium sulphate solution and 10 cc. of 10% barium acetate solution. Collect the precipitate in a compact mass (a centrifuge may be used advantageously) and wash upon a small filter. Transfer to a platinum crucible and ignite.

Dip a carefully cleaned glass plate, while hot, in a mixture of equal parts of Carnauba wax and paraffin and allow to cool. Make, with a sharp instrument, a distinctive mark through the wax, taking care not to scratch the surface of the glass.

Add a few drops of concentrated sulphuric acid to the residue in the crucible and cover with the waxed plate, having the mark nearly over the center and making sure that the edge of the crucible is in close contact with it. Keep the top surface of the plate cool by means of a suitable device and heat the crucible for an hour at as high a temperature as practicable without melting the wax (an electric stove gives the most satisfactory form of heat).

If fluorids be present, a distinct etching will be apparent on the glass where it was exposed.

25

Method II.

The preceding method may be varied by mixing a small amount of precipitated silica with the precipitated barium fluorid and applying the method for the detection of fluosilicates, under 27 or 28.

This method is of value in the case of foods whose ash contains a considerable amount of silica. Under these circumstances, concentrated sulphuric acid liberates silicon fluorid, which would escape detection under 24.

FLUOBORATES AND FLUOSILICATES.

26

PREPARATION OF SAMPLE.

Make about 200 grams of the sample alkaline with lime water, evaporate to dryness, and incinerate. Extract the crude ash with water, to which sufficient acetic

acid has been added to decompose carbonates, filter, ignite the insoluble portion, extract with dilute acetic acid, and again filter. The insoluble portion now contains calcium silicate and fluorid, while the filtrate will contain all the boric acid present.

QUALITATIVE TESTS.

27

Method I.¹⁰

Incinerate the filter, from 26, containing the insoluble portion, mix with a little precipitated silica, transfer to a short test tube, attached to a small U-tube containing a few drops of water and add 1-2 cc. of concentrated sulphuric acid. Keep the test tube in a beaker of water on the steam bath for 30-40 minutes. If any fluorin be present, the silicon fluorid generated will be decomposed by the water in the U-tube and will form a gelatinous deposit on the walls of the tube.

Next test the filtrate as directed under 14. If both hydrofluoric and boric acids be present, it is probable that they are combined as borofluorid. If, however, silicon fluorid is detected and not boric acid, the operation should be repeated without the introduction of the silica, in which case the formation of the silicon skeleton is conclusive evidence of the presence of fluosilicate. In an ash containing an appreciable amount of silica, sulphuric acid will liberate silicon fluorid rather than hydrofluoric acid. The presence of a fluosilicate is indicated, therefore, and not the presence of a fluorid.

28

Method II.

Incinerate the filter, from 26, containing the insoluble portion, in a platinum crucible, mix with a little precipitated silica, and add 1 cc. of concentrated sulphuric acid. Cover the crucible with a watch glass, from the underside of which a drop of water is suspended, and heat for an hour at 70°-80°C., keeping the watch glass cooled. The silicon fluorid which is formed is decomposed by the water, leaving a gelatinous deposit of silica and etching a ring at the periphery of the drop of water. Test the filtrate for boric acid as directed under 14.

SULPHUROUS ACID.

29

Qualitative Test.¹¹

Add some sulphur-free zinc, and several cc. of hydrochloric acid to about 25 grams of the sample (with the addition of water, if necessary) in a 200 cc. Erlenmeyer flask. In the presence of sulphites, hydrogen sulphid will be generated and may be detected with lead acetate paper. Traces of metallic sulphids are occasionally present in vegetables, and will give the same reaction as sulphites under the conditions of the above test. Positive results obtained by this method should be verified by the distillation method under 30.

It is always advisable to make the quantitative determination of sulphites, owing to the danger that the test may be due to traces of sulphids. A trace is not to be considered sufficient indication of the presence of sulphur dioxid either as a bleaching agent or as a preservative.

TOTAL SULPHUROUS ACID.

30

Method I.—Distillation Method.

Distil 20-100 grams of the sample (adding recently boiled water if necessary) in a current of carbon dioxid, after the addition of about 5 cc. of a 20% glacial phos-

phoric acid solution, until 150 cc. have passed over. Collect the distillate in about 100 cc. of nearly saturated bromin water, allowing the end of the condenser to dip below the surface. The method and apparatus may be simplified without material loss in accuracy by omitting the current of carbon dioxid, adding 10 cc. of phosphoric acid instead of 5 cc., and dropping into the distillation flask, immediately before attaching the condenser, a piece of sodium bicarbonate weighing not more than 1 gram. The carbon dioxid liberated is not sufficient to expel the air entirely from the apparatus, but will prevent oxidation to a large extent. When the distillation is finished, boil off the excess of bromin, dilute the solution to about 250 cc., add 5 cc. of hydrochloric acid (1 to 3), heat to boiling, and precipitate the sulphuric acid with 10% barium chlorid solution. Boil for a few minutes longer, allow to stand overnight in a warm place, filter on a weighed Gooch, wash with hot water, ignite at a dull red heat, and weigh as barium sulphate.

31

Method II.—Direct Titration Method.

In the examination of wine, fairly accurate results may be obtained by the following method:

Place 25 cc. of 5.6% potassium hydroxid solution in a 200 cc. flask. Introduce 50 cc. of the sample, mix with the potassium hydroxid solution, and allow the mixture to stand for 15 minutes with occasional agitation. Add 10 cc. of sulphuric acid (1 to 3) and a few cc. of starch solution, and titrate the mixture with N/50 iodine solution. Introduce the iodine solution as rapidly as possible and continue the addition until the blue color persists for several minutes. One cc. of N/50 iodine is equivalent to 0.00064 gram of sulphur dioxide.

DETERMINATION OF FREE SULPHUROUS ACID.

32

(Especially Adapted to Wine.)

Treat 50 cc. of the sample in a 200 cc. flask with about 5 cc. of sulphuric acid (1 to 3) add about 0.5 gram of sodium carbonate to expel the air, and titrate the sulphurous acid with N/50 iodine, as directed under 31.

BETA-NAPHTHOL.

33

Qualitative Test.

Extract 200 cc. of the sample, or of its aqueous extract, prepared as directed under 1 (C), with 10 cc. of chloroform in a separatory funnel. To the chloroform extract in a test tube add a few drops of alcoholic potash, and place in a boiling water bath for 2 minutes. The presence of beta-naphthol is indicated by the formation of a deep blue color, which changes to green and then to yellow.

ABRASTOL.

QUALITATIVE TESTS.

34

Sinibaldi Method.¹³

Make 50 cc. of the sample alkaline with a few drops of ammonium hydroxid and extract with 10 cc. of amyl alcohol, adding ethyl alcohol if an emulsion is formed. Decant the amyl alcohol, filter if turbid, and evaporate to dryness. Add to the residue 2 cc. of nitric acid (1 to 1), heat on the water bath until half of the liquid is evaporated, and transfer to a test tube with the addition of 1 cc. of water. Add about 0.2 gram of ferrous sulphate and an excess of ammonium hydroxid, drop by

drop, with constant shaking. If the resultant precipitate is of a reddish color, dissolve it in a few drops of sulphuric acid, and add ferrous sulphate and ammonium hydroxid as before. As soon as a dark colored or greenish precipitate is obtained, introduce 5 cc. of alcohol, dissolve the precipitate in sulphuric acid, shake well and filter. In the absence of abrastol this method gives a colorless or light yellow liquid, while a red color is produced in the presence of 0.01 gram of abrastol.

35

*Sanglé-Ferrière Method.*¹³

Boil 200 cc. of the sample with 8 cc. of concentrated hydrochloric acid for an hour in a flask fitted with a reflux condenser. Abrastol is thus converted into betanaphthol and is detected as directed under 33.

SUCROL OR DULCIN.

QUALITATIVE TESTS.

36

*Morpurgo Method.*¹⁴

Evaporate about 100 cc. of the sample, or of the aqueous extract prepared as directed under 1 (C) and neutralized with acetic acid, to a sirupy consistency after the addition of about 5 grams of lead carbonate, and extract the residue several times with 90% alcohol. Evaporate the alcoholic extract to dryness, extract the residue with ether, and allow the ether to evaporate spontaneously in a porcelain dish. Add 2 or 3 drops each of phenol and concentrated sulphuric acid and heat for about 5 minutes on the water bath, cool, transfer to a test tube and overlay with ammonium hydroxid or sodium hydroxid solution with the least possible mixing. The presence of dulcin is indicated by the formation of a blue color at the zone of contact.

37

*Jorissen Method.*¹⁵

Suspend the residue from the ether extract obtained as directed above in about 5 cc. of water; add 2-4 cc. of an approximately 10% solution of mercuric nitrate, and heat for 5-10 minutes on the water bath. In the presence of sucrol a violet blue color is formed, which is changed to a deep violet on the addition of lead peroxid.

FORMIC ACID.

*Quantitative Method.*¹⁶

38

REAGENTS.

(a) *Sodium acetate solution.*—Dissolve 50 grams of dry sodium acetate in sufficient water to make 100 cc. and filter.

(b) *Mercuric chlorid reagent.*—Dissolve 100 grams of mercuric chlorid and 150 grams of sodium chlorid in sufficient water to make 1 liter and filter.

(c) *Tartaric acid.*

(d) *Barium carbonate.*

39

APPARATUS.

The apparatus required (Fig. 6) consists of a steam generator (S), a 300 cc. flask (A) in which the sample is placed, a 500 cc. flask (B), containing a suspension of barium carbonate, a spray trap (T), a condenser, and a 1 liter graduated flask (C). The tip of the tube (D), leading into (B), consists of a bulb containing a number of small holes to break the vapor into small bubbles.

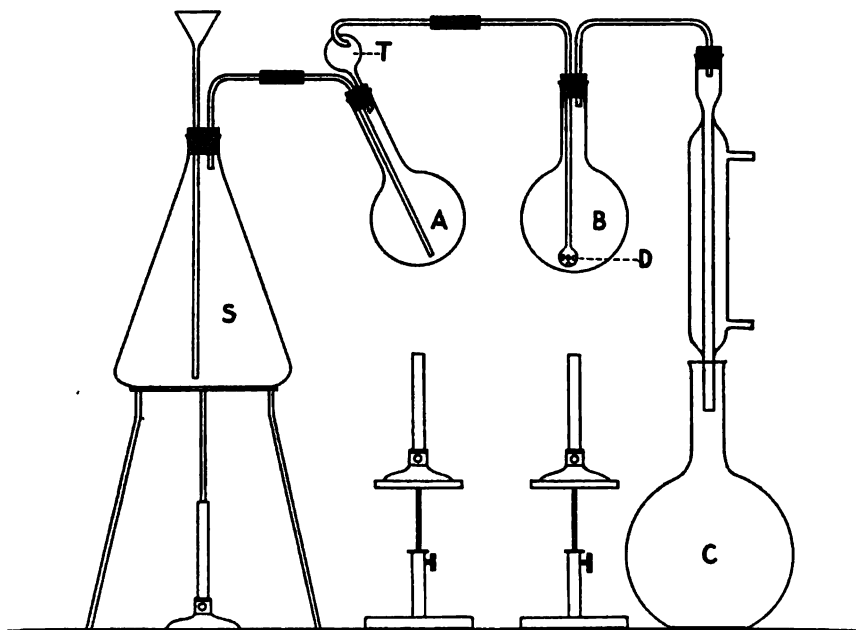


FIG. 6. APPARATUS FOR DETECTION OF FORMIC ACID.

40

DETERMINATION.

For thin liquids like fruit juices, use 50 cc. For heavy liquids and semi-solids like sirups and jams, use 50 grams diluted with 50 cc. of water. Place the sample in the flask (A), add 1 gram of tartaric acid, and connect as shown in Fig. 6, the flask (B) having been charged previously with a suspension of 2 grams of barium carbonate in 100 cc. of water. If much acetic acid is present, sufficient barium carbonate must be used so that at least 1 gram remains at the end of the operation. Heat the contents of flasks (A) and (B) to boiling and distil with steam from the generator (S), the vapor passing first through the sample in flask (A), then through the boiling suspension of barium carbonate in (B), after which it is condensed, and measured in the graduated flask (C). Continue the distillation until 1 liter of distillate is collected, maintaining the volume of the liquids in the flasks (A) and (B) as nearly constant as possible by heating with small Bunsen flames, and avoiding charring of the sample in the flask (A). After 1 liter of distillate has been collected, disconnect the apparatus and filter the contents of flask (B) while hot, washing the barium carbonate with a little hot water. The filtrate and washings should now measure about 150 cc. If not they should be boiled down to that volume. Then add 10 cc. of the sodium acetate, 2 cc. of 10% hydrochloric acid, and 25 cc. of the mercuric chlorid reagent. Mix thoroughly and immerse the container in a boiling water bath or steam bath for 2 hours. Then filter on a tared Gooch, wash the precipitate thoroughly with cold water and finally with a little alcohol. Dry in a boiling water oven for 30 minutes, cool, weigh, and calculate the weight of formic acid present by multiplying the weight of the precipitate by 0.0975. If the weight of mercurous chlorid obtained exceeds 1.5 grams, the determination must be repeated, using more mercuric chlorid reagent or a smaller amount of sample. A blank

test should be conducted with each new lot of reagents employed in the reduction, using 150 cc. of water, 1 cc. of 10% barium chlorid solution, 2 cc. of 10% hydrochloric acid, 10 cc. of the sodium acetate, and 25 cc. of the mercuric chlorid reagent, heating the mixture in a boiling water bath or steam bath for 2 hours. The weight of mercurous chlorid obtained in this blank test must be deducted from that obtained in the regular determination.

BIBLIOGRAPHY.

- ¹ J. Ind. Eng. Chem., 1910, 2: 24.
- ² Z. Nahr. Genussm., 1910, 19: 137; C. A., 1910, 4: 1523.
- ³ U. S. Div. Chem. Bull. 51, p. 113.
- ⁴ Sutton. Volumetric Analysis. 10th ed., 1911, p. 95.
- ⁵ Z. Nahr. Genussm., 1902, 5: 353.
- ⁶ Analyst, 1895, 20: 155.
- ⁷ Ann. di farmacoterapia e chim., 1898, 27: 97; Chem. Zentr., 1898, (1), 1152; 1902, (1), 1076; J. Soc. Chem. Ind., 1898, 17: 697; Chem. Ztg., 1902, 26: 246; Abs. J. Chem. Soc., 1902, 82: 367.
- ⁸ Service de Surveillance des Aliments en Belgique, through Bul. soc. chim. belg., 1897-8, 11-12: 211; Abs. Analyst, 1897, 22: 282.
- ⁹ Chem. News, 1905, 91: 39; Ann. Rept. Mass. State Board of Health, 1905, p. 498.
- ¹⁰ Mon. Sci., 1895, (4), 9: 324.
- ¹¹ U. S. Div. Chem. Bull. 13, (8), p. 1032.
- ¹² Mon. Sci., 1893, (4), 7: 842.
- ¹³ Compt. rend., 1893, 117: 796.
- ¹⁴ Z. anal. Chem., 1896, 35: 104.
- ¹⁵ Ibid., 628.
- ¹⁶ Biochem. Z., 1913, 51: 253.

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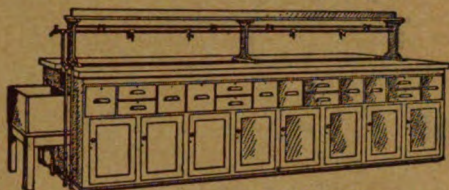
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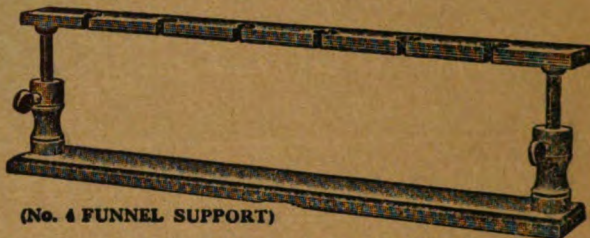


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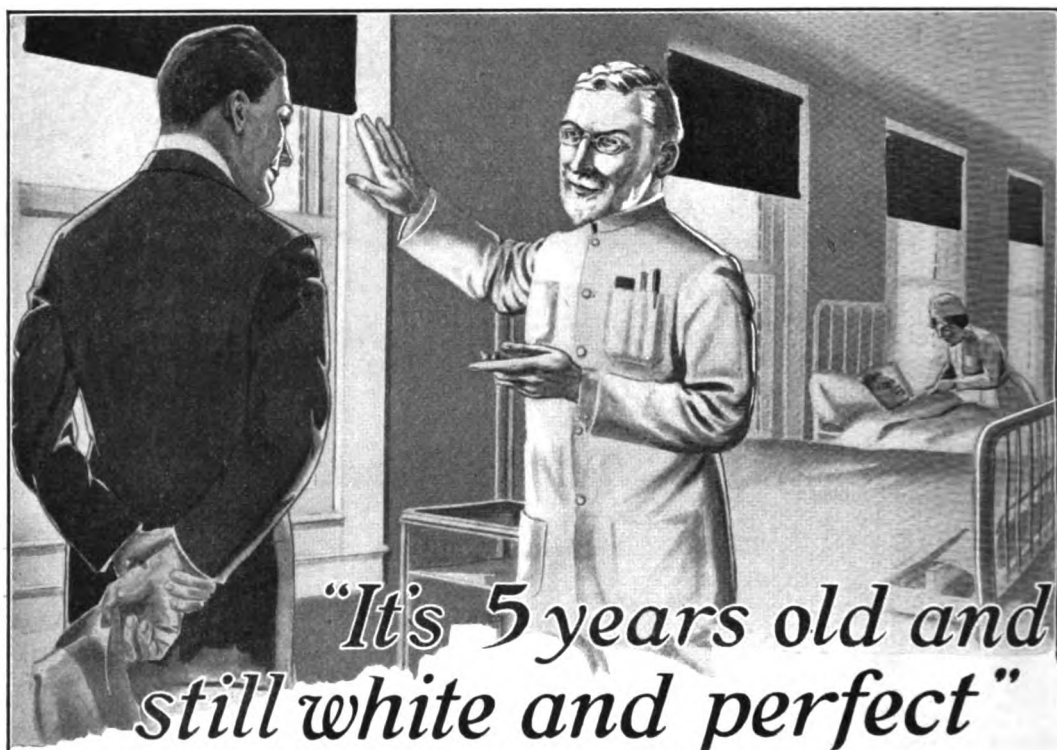
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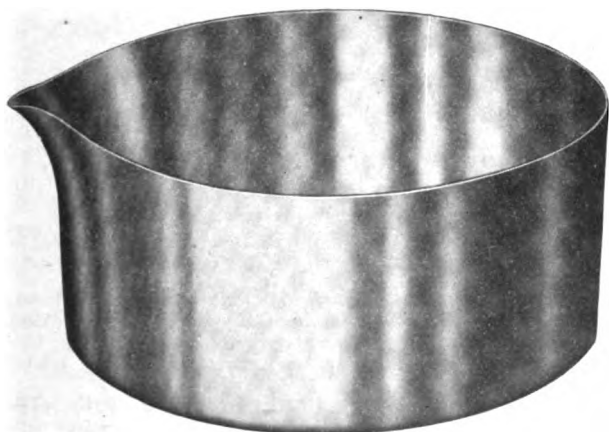
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RESULT—Average breaking temperature of twenty 400 cc. Pyrex Beakers, 253°C.

The beakers tested varied in weight from 83 to 114 grams each, the average being 94.5 grams. The minimum breaking temperature was 230°C. and the maximum 280°C.

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The method employed is that standardized by P. H. Walker for comparing the stability of glasses intended for laboratory purposes (see *Journal of the American Chemical Society*, Vol. 27, Page 865, 1905). Beakers of 250 cc. size with 150 cc. redistilled water were immersed in steam bath 48 hours, water being added to keep the volume constant. The method used differed from Walker in that Walker titrates the alkali in the resulting solution, while in these tests the solution was evaporated to dryness in a small platinum dish and the total amount dissolved weighed. Deductions of 0.0001 to 0.0003 are made for non-volatile impurity in the water.

RESULT—Average solubility toward distilled water for each 100 square centimeters of exposed area in Pyrex ware, 0.00015 grm.

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Starting at six inches the beaker is dropped right side up and as nearly level as possible on a two-inch pine plank firmly supported, and this is repeated increasing the height of drop two inches at a time. To avoid excessive bounding and possibility of chipping the upper edge, a piece of cloth is laid around the spot on which the beaker is dropped.

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PRESIDENT'S ADDRESS.¹

THE STORAGE OF SUGAR IN THE MAPLE TREE.

By C. H. JONES (Agricultural Experiment Station,
Burlington, Vt.), *President*.

Maple sugar is obtained mainly from the species of maple known to botanists as *Acer saccharum*. Census returns indicate a total yearly production in the United States of the equivalent of some 45,000,000 pounds. The more northern sections contribute most largely to the supply. The yield is subject to extreme variations, being dependent on certain daily weather conditions during a few weeks in early spring. Although insignificant in amount when compared with the world's production of cane and beet sugars, yet as a table luxury maple products have long held an honored place among our food products.

ECONOMIC INVESTIGATIONS.

Economic investigations connected with the production of maple sugar and sirup have occupied the attention of many students of the subject and their observations have been recorded in various publications of a scientific and popular nature. They include data on the history of the maple industry, composition and flow of maple sap, the action of micro-organisms on sap and their effect on the finished product, together with a vast amount of strictly chemical data directed toward a proper standardization of the marketable product and the detection of adulteration therein.

Chemically speaking, the sugar from the maple has been shown to be identical in composition with the ordinary granulated sugar secured from the cane and beet. The importance it holds as a delicacy is due to a characteristic and pleasing flavor associated with it which, however, is destroyed if the usual methods of sugar refining are applied to the crude product.

The nature of the considerations about to be presented is fundamental rather than economic; fundamental in that they deal with the tree itself and seek to indicate the various changes taking place within the trunk at different seasons of the year and their visible manifestations as indicated by pressure and sap flow. Before proceeding further a brief description of maple wood will be given. No attempt is made to describe the several structures in detail but attention is directed to such tissues as seem most directly concerned with our general subject.

¹ Presented Tuesday, November 16, as special order of business for 11.30 o'clock.

GENERAL STRUCTURE.

In general structure, the maple trunk or stem (*xylem*) may be divided into a brown heart wood (*duramen*) and a white sap wood (*alburnum*). The sap wood contains the living cells and is the main conducting medium for water and food. The heart wood is relatively lifeless and functions largely as a mechanical support for the crown and as a reservoir in which to store water during the winter months.

The stem is composed of three kinds of cells, viz., wood fibers, wood parenchyma and vessels, without intercellular spaces. At right angles to these cells, extending from the circumference toward the heart, are the *medullary rays*. These are *living cells*. They vary greatly in width and length and serve as the main centers for the storage of starch. Measurements of the medullary rays in the maple have shown that they occupy about 18% by volume of the outer or recently formed sap wood.

The starch grains found in the maple are angular, not uniform in shape, and vary in size from 1.4–5.7 microns in diameter. The characteristic blue color is obtained by treatment with iodine solution. Starch is stored not only in the medullary rays but also in the wood (parenchyma) which in some cases forms pockets, visible to the naked eye. These as a rule extend at right angles to and border on the medullary rays. Starch may also be found in the rows of cells (from three to many) formed in the summer wood of the past few years.

Between the medullary rays are longitudinal cells often of great length containing water and food ready for transference throughout the tree. They are known as *tracheae*. Associated with these are similar but shorter cells known as *tracheids*. Both *tracheae* and *tracheids* are *dead cells*. They comprise about 7% by volume of the sap wood.

CHEMICAL PHASES OF SUGAR STORAGE.

A consideration of the more strictly chemical phases of sugar storage in the maple will indicate conditions existing within the tree during the year. The specimens used in determining this matter were second growth maples, quite uniform as to size, age and leaf area, which grew near each other, under similar general conditions of soil formation and exposure. Differences due to these causes were thus minimized and as samples were taken monthly for 29 months a period is represented including 3 successive sugar seasons, 2 full periods of growth and 3 periods of rest. To further confirm these observations precautions were taken during the second 12 months to secure 2 trees on the dates which the first year's work indicated to be what may be termed critical points in the yearly cycle.

The several trees were felled, measured and immediately brought to

the laboratory where suitable sections representing the base (outer and inner wood) and the top wood were reduced to sawdust by means of a circular saw. The samples thus prepared were subjected to chemical analysis directed primarily to ascertain the content of moisture, sugar, and starch, which are assumed to be the so-called readily available stored foods. The last transformation of the sugar molecule used for plant food purposes is outside a strictly chemical province, being rather a problem for the plant physiologist.

While variations in the results obtained for corresponding months in different years were noted, and individual differences were occasioned by samples and natural fluctuations in soil moisture, sunlight and temperature conditions, yet the same general trend of all results was plainly manifested during successive seasons.

Instead of burdening you with a mass of data representing monthly conditions for 29 months the results have been summarized in a brief table where the months of the year are divided into 4 groups, each group representing a distinct epoch in the life functions of the tree.

The first division includes the months of December, January and February, the winter months, during which interval the tree is generally considered dormant as regards development. The second division comprises the months of March and April. The sap flow occurs during this

TABLE 1.
Percentage of available carbohydrate food in maple wood.

DETERMINATION	DECEMBER, JANUARY AND FEBRUARY	MARCH AND APRIL	MAY, JUNE, JULY AND AUGUST	SEPTEMBER, OCTOBER AND NOVEMBER
OUTER BASE WOOD:				
Water.....	35.01	32.86	30.30	29.80
Sucrose.....	0.94	0.80	0.86	0.72
Reducing sugars.....	1.40	0.53	0.13	0.41
Starch.....	0.78	0.96	1.01	1.43
INNER BASE WOOD:				
Water.....	30.28	29.64	26.87	29.05
Sucrose.....	0.49	0.52	0.18	0.41
Reducing sugars.....	0.65	0.33	0.09	0.26
Starch.....	0.43	0.49	0.62	0.51
TOP WOOD (outer and inner):				
Water.....	33.85	31.36	32.46	32.14
Sucrose.....	1.12	1.04	0.41	0.83
Reducing sugars.....	1.79	0.67	0.23	0.59
Starch.....	1.09	1.33	1.23	1.63
ROOTS:				
Water.....	33.07	35.46	31.00	30.23
Sucrose.....	1.52	0.93	0.66	1.41
Reducing sugars.....	1.00	0.79	0.42	0.32
Starch.....	3.92	2.25	3.41	5.76

period which, in northern latitudes, is designated the sugar season. The tree is preparing for its yearly work of growth and reproduction. The third period, May, June, July and August, represents the time of bud and leaf development, the formation of the annual ring and the storage of reserve food. The fourth division, September, October and November, is a period of gradual preparation for the cold of winter. Early in this period, as the leaves fall, reserve foods are deposited largely in the form of starch which later may be changed in part to sugar.

SUCROSE IN TRACHEAE AND TRACHEIDS.

When fresh maple wood is taken from a tree, for example during March or April, suitably pulverized, and treated with water, the resulting solution is found to contain both reducing sugars and sucrose. Maple sap as it flows from the tree has been shown repeatedly to be practically free from reducing sugars, but to contain sucrose. This would indicate that there is a separation of the two sugar solutions in the tree.

By taking a suitable section of maple wood and subjecting it to a water pressure of about 30 pounds, it was found that only sucrose was removed, no reducing sugars being washed out. The amount of sucrose thus obtained depended to some extent on the quantity of water forced through the stem. Three trials on different samples using 1250, 900 and 2500 cc. of water removed 28, 51 and 67% respectively of the total sucrose present. It would seem, therefore, that sucrose must be stored chiefly in the *tracheae*, the long longitudinal tubes, to which mention has been made, while the reducing sugars must be contained in the cell sap of the living cells.

CONSIDERATION OF RESULTS.

From a study of Table 1 it is clear that marked variations exist in the water, sugar and starch content of wood in different parts of the maple tree at similar periods and also that the composition is not constant during the year. Further, that the top wood is supplied more abundantly with sugars and starch than either the outer or inner base wood.

Considering the results obtained in the periods indicated, it is noted that the amount of water present is greatest in the outer, inner and top wood sections during the cold winter months, December to February. Similarly, the sugars are also higher. The starch, on the other hand, is less at this time than at other intervals during the year. The percentage of water is lowest in the inner wood but if account is taken of the total bulk of wood represented a large excess of water would be found in the inner, over that contained in the outer, base tissues.

The March-April period shows a slight reduction in the water content of the several sections. The sugar percentages continue to decrease as warmer weather approaches, while the starch content increases.

The third period, representing the season of growth, indicates that the amount of water in the tree is at its minimum, large amounts having been given off by transpiration. The percentage amount of sugars present is small but it remains quite uniform during the growing season. The starch content is practically unchanged in the outer and top wood, while the inner tissues show an increase. The small amount of sugars present during this period of active photosynthesis is explained first, by the fact that these constituents have been drawn on to feed the developing buds and leaves and, secondly, by the fact that the elaborated food is used immediately in the development of the annual ring.

The last period, September to November, finds the water content of the outer and top wood remaining constant while a decided gain in moisture is observed in the inner section. The sugars show an increase of some 130% over amounts present during the growing season. In like manner the starch percentages have become augmented in the outer and top sections.

The roots exhibit the highest water content in March and April, the highest sugar content from December to February and the lowest between May and August. The starch content of the roots greatly exceeds that found in the trunk wood regardless of season. It is most abundant during the autumn months, September to November, when an average percentage of 5.76 was obtained. This gradually decreases during the cold winter months and is lowest during the period just preceding the renewal of spring activities when but 2.25% was indicated.

RELATION OF SUCROSE TO REDUCING SUGARS.

There is a condition shown by the results in Table 1 to which special attention should be directed and that is the relation between the percentages of sucrose and reducing sugars and the relation of both to the starch content. Reducing sugars are greater in amount than is sucrose during only one period of the year, December to January. Sucrose predominates throughout the remaining months. The change in relativity begins just previous to the sugar season, March and April. Coincident with the decrease in reducing sugars an increase in starch percentage is observed. The roots throughout the year show a predominance of sucrose over reducing sugars, particularly in the autumn months of September, October and November. Mention already has been made of the large starch content.

DENSITY OF SAP SOLUTIONS.

Investigators are most familiar with the composition of maple sap, secured during March and April by the ordinary tapping process, from that part of the tree designated in Table 1 as outer base wood. It is

commonly known that maple sap is essentially a dilute solution of sucrose containing small amounts of mineral matter and that its sugar content varies from 1-7% with an average of approximately 3%. Reference to Table 1 shows that but 0.80% of sucrose was present in the basewood. This amount, however, must have been held in solution by the water in the tree trunk, as were the reducing sugars, and a simple calculation will show the sucrose content of the sap to have been 2.34%. This represents an average for several young trees during 3 successive sugar seasons.

The grouping of averages by periods in Table 1 does not emphasize the high concentrations obtained on certain dates. Thus, when the individual tree data are consulted, instances are not lacking which show that this concentration during the sugar season ran as high as 5% of sucrose and that even a total sugar concentration of 12% occurred during extremely cold weather.

In order that the density of the sugar solutions found in the tree may be readily compared the results already tabulated have been calculated to a concentration or density basis.

TABLE 2.
Percentage density of sugar solutions in maple wood.

DETERMINATION	DECEMBER, JANUARY AND FEBRUARY	MARCH AND APRIL	MAY, JUNE, JULY AND AUGUST	SEPTEMBER, OCTOBER AND NOVEMBER
OUTER BASE WOOD:				
Sucrose.....	2.52	2.34	1.17	2.33
Reducing sugars.....	3.75	1.55	0.42	1.33
INNER BASE WOOD:				
Sucrose.....	1.56	1.71	0.66	1.38
Reducing sugars.....	2.07	1.08	0.33	0.87
TOP WOOD (outer and inner):				
Sucrose.....	3.05	3.14	1.24	2.47
Reducing sugars.....	4.87	2.03	0.69	1.76
Roots:				
Sucrose.....	4.27	2.50	2.06	4.41
Reducing sugars.....	2.81	2.12	1.31	1.00

The same general variations already discussed are again in evidence but these figures more nearly represent the density of the sucrose and reducing sugar solutions found in the cells and tissues composing the tree structure. Starch, being insoluble in water, is not included in this calculation although one would be justified in so including it for it is well known that starch is one of the reserve foods stored by the maple and that it must be transformed into soluble sugars before it can be transported.

DENSITY OF SAP INCREASES FROM BASE TO TOP OF TREE.

The ascent of water and food to the tops of tall trees has long occupied the attention of scientists. No explanation, entirely satisfactory, thus far has been presented although many theories have been advanced. Data as to the amounts of soluble sugars and water in different parts of the tree undoubtedly have a bearing on this question. The increased carbohydrate concentration in the top wood of the maple over that in the base section has been noted. For several reasons, particularly when the structure of the wood and region of consumption of food are considered, it seems logical to assume that this increase in concentration of the sugars, together with the higher starch content, should be a gradual one, as we ascend the tree.

A small maple tree cut early in May was examined in sections of 4 feet each, from the base to the top, with the following results:

TABLE 3.
Percentage density of sugar solutions.

SECTION	WATER	STARCH	REDUCING SUGARS	SUCROSE	TOTAL SUGARS
1st (base).....	29.07	0.83	0.10	1.82	1.92
2nd.....	33.15	0.81	0.21	1.78	1.99
3rd.....	28.97	0.92	0.37	2.26	2.63
4th.....	26.85	1.06	0.32	2.92	3.24
5th (top).....	25.84	1.10	0.30	3.10	3.40

Table 3 would seem to indicate that the sugar concentration of both active cell, and *tracheae*, sap gradually increases from the lower portions of the tree trunk upward. Starch also shows a similar increase. On the other hand, water, in this instance, decreases in amount as we ascend.

INTERPRETATION OF RESULTS.

It is a relatively easy matter to present for consideration data secured by physical and chemical operations in the field and laboratory but the significance of such data when applied to the solution of nature's processes is not so readily formulated. A lengthy interpretation of the results bearing on the phenomena of sugar formation and sap flow will not be attempted but a few general deductions enumerated.

The work of numerous investigators has shown clearly that sunlight, leaf area and soil moisture are factors influencing the abundant storage of starch and sugars. A correlation table, showing relationships between stored carbohydrates on the one hand, and sunlight, leaf area and rainfall on the other, undoubtedly would give most suggestive results. It is a different matter to prove the relationship from a practical or economic standpoint as weather conditions at the time of sap flow seriously affect

results. In other words, one can determine with reasonable certainty that an abundance of carbohydrates has been stored in the tree but the best of prophecies fail when attempts are made to predict the amount of sugar likely to be secured the following spring.

The yearly life cycle of the maple tree has for its objects nutrition and reproduction. Green leaves manufacture starch daily in sunlight. At night it is transformed to soluble carbohydrates and carried down the inner bark and cambium layer where it is again deposited in the medullary rays as starch or remains in solution in the tree tissues. The excess only is thus deposited, what is necessary being used for the formation of the annual ring.

When the leaves fall in the autumn a protective enzyme action may take place in the medullary rays, and sucrose is ultimately formed and distributed through the *tracheae*. The increased concentration of the sugar solutions in the maple trunk during the cold weather period appears to have for its purpose the protection of the tree tissue from freezing, so that an early transference of abundant water and food materials to the leaf-bearing sections of the tree may be established. The maple is the earliest of northern trees to develop its buds and leaves in spring.

The rate of movement of sap in the tapped tree has been measured by injecting lithium chlorid and noting the time of its appearance in the sap from holes at known distances above and below the point of insertion. A rate of 2-6 inches per minute was observed. Since this flow is an artificial one, stimulated nevertheless by conditions within the tree, it does not follow that sap movement up and down within the untapped tree trunk, before leaf formation has started, parallels these rates. Doubtless the water movement is far less rapid, although there is warrant for asserting that changes in sugar concentration and water content in different sections of the tree are taking place constantly.

Considerable work has already been done touching the matter of direction of sap flow and pressure under the artificial conditions created by the ordinary tapping process. It has been shown that during certain weather conditions in early spring, characterized by a sharp frost at night and a rise in temperature to a few degrees above the freezing point the following day, a state of pressure is manifested in the tree trunk and sap is exuded at the tap hole or point of least resistance. This pressure may vary from 1-25 pounds and while the flow of sap is not necessarily in direct proportion to the pressure, there is strong evidence nevertheless that some relation exists. During the greater part of the year a state of negative pressure or suction is exerted by the tree.

The simple determination of the water content of maple wood at different seasons of the year is not without interest. It is more than a coincidence and doubtless for a definite purpose that the highest water

content always precedes the leaf formation and transpiration period and that the lowest amount is present when the leaves have ceased their activities.

Beginning in December, the reducing sugars exceed the sucrose content of maple wood until March when a decided decrease occurs in the percentage of reducing sugars. A relatively high percentage of sucrose obtains until May. A predominance of sucrose over reducing sugars, though in reduced amounts, is maintained through the summer and fall, until, in December, the reducing sugars again take the lead. Thus a high sugar content is found during the coldest period, reducing sugars predominating over sucrose, the ratio of reducing sugars, starch and sucrose being 1.5 : 0.8 : 1. As warm weather approaches the total sugar content decreases and a most pronounced increase of sucrose over reducing sugars occurs, the above ratio becoming 0.7 : 1.2 : 1.

Warm weather favors the deposition of starch from soluble carbohydrates. Continued cold weather causes the insoluble starch to decrease in amount, transforming it to soluble carbohydrates, thus increasing the total sugar content during the winter months.

From the chemical evidence presented it is plain that changes are taking place within the tree throughout the year. A fall deposition of starch occurs. Later, as cold weather approaches, the leaf-manufactured food that has been deposited as starch is changed in the living protoplasmic cells to reducing sugars and sucrose which, held in a water solution, fill the vessels throughout the tree. These sugars are most abundant in the young living tissues of the trunk nearest the cambium layer.

Reducing sugars and starch seem to be most intimately associated with the living protoplasmic cells, while a considerable portion at least of the elaborated sucrose fills the dead vessels. These channels, it is well established, serve as quick carriers for water and sucrose to upper and distant parts of the tree, and are in direct communication with the roots or water-gathering system. The uniformity of sap concentration during the four months comprising the growing season indicates that the leaf elaborated carbohydrates are used, either directly or indirectly, for the development of the annual ring. Until that duty is performed no marked increase in soluble carbohydrate storage is noted.

OUR JOURNAL.

In conclusion I desire to say a few words on the subject of our new JOURNAL. The problems confronting us and the ways and means for dealing with them were ably considered by my predecessor in his annual address one year ago. As you all know this association now has a quarterly publication and our deliberations, printed in a most attractive form,

will soon be placed promptly at our disposal. This publication is not a one man volume and every member should be allowed and encouraged to do his share. It is needless for me to urge the members to subscribe but I do want to ask particularly that the younger members not only subscribe but also read and study the JOURNAL with a view toward detecting shortcomings and rectifying them in the future.

It is too early to predict the ultimate outcome of our venture. We have men enough, brains enough and, I hope, a loyalty to our chosen profession sufficient to carry on this project.

JOURNAL

OF THE

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PROCEEDINGS OF THE THIRTIETH ANNUAL CONVENTION.

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VI. LEATHERS.—TENTATIVE.

VEGETABLE TANNED LEATHER.

1

PREPARATION OF SAMPLE.

Grind the sample, without undue heating, and pass through a 10 mesh sieve. The ground sample must not contain hard lumps. Plane heavily greased leathers (containing more than 20 % fat) into very thin shavings. Spread out the prepared sample and allow it to return to atmospheric moisture condition; mix thoroughly, and place in tightly covered containers.

2

MOISTURE.

Place 10 grams of the sample, as prepared under 1, in a tared, wide, shallow, weighing bottle (or a similar dish which can be covered tightly), and dry in a water oven for 15 hours at 98°–100°C. Cover the weighing bottle, cool in a desiccator containing sulphuric acid, and weigh. The moisture present in the leather as received may be determined by cutting it quickly into small pieces and drying without grinding as directed above.

3

TOTAL ASH.

Incinerate slowly 5 grams of the sample, as prepared under 1, at a dull red heat. If difficulty is experienced in burning off the carbon, leach the residue with hot water, filter on an ashless filter, dry and ignite the filter and residue, add the filtrate, evaporate to dryness and ignite. Cool in a desiccator containing sulphuric acid and weigh.

The ash may be examined for acids and bases by any suitable method. Aluminium, magnesium, sodium, barium, calcium and lead are the bases, and hydrochloric and sulphuric acids are the acids which it may be necessary to determine.

4

INSOLUBLE ASH.

Incinerate slowly the residue from the extraction of water-soluble material, obtained in 6 or 7, until all the carbon is burned off, cool in a desiccator containing sulphuric acid and weigh.

5

FATS.

Place, without packing, 15 grams of the leather, as prepared under 1, in a Soxhlet or Johnson extractor with a layer of fat-free cotton above and below the sample. Extract 8–10 hours with petroleum ether distilling between 50° and 80°C. Heavily greased leathers (containing 15% or more fat) will require the maximum time. Remove the receiving flask, evaporate the petroleum ether on the steam bath and dry the fat residue for 3 hours in a water oven at 98°–100°C., cool in a desiccator and weigh. Repeat the drying in the water oven for periods of 1–1½ hours, cooling and weighing as before, until no further loss in weight occurs. Retain the leather residue from the fat extraction for the extraction of water-soluble material in 6 or 7.

EXTRACTION OF WATER-SOLUBLE MATERIAL.

6

Method I.

Evaporate the petroleum ether from the fat-free leather, obtained under 5, and moisten thoroughly with from 100–150 cc. of water. Place a layer of cotton in the

bottom of a Soxhlet extractor designed for making extractions at temperatures below 100°C.

An extractor of this kind is furnished with a water jacket surrounding that portion of the apparatus containing the sample but does not enclose the side tube which carries the hot vapors to the condenser.

Transfer the moistened fat-free leather to the extractor, and cover this with another layer of cotton to avoid siphoning off solid particles. Maintain the temperature of the jacket surrounding the Soxhlet at 50°C. (1) Pour 200 cc. of water (including that used in moistening the leather) into the Soxhlet and allow it to siphon into the flask below, then heat and extract for an hour. Remove the flame and transfer the extract to a liter graduated flask. Then add water and continue the extraction as directed below, removing and transferring the extract to the liter flask before each fresh addition of water.

(2) Add 175 cc. of water and extract for 2 hours.

(3) Add 175 cc. of water and extract for 3 hours.

(4) Add 175 cc. of water and extract for 4 hours.

(5) Add 175 cc. of water and extract for 4 hours.

Transfer the last portion of the extract to the graduated flask. This gives 14 hours' extraction and an extract which does not exceed 1 liter in volume. Dilute to 1 liter at room temperature and mix thoroughly.

7

Method II.

(This method is the same in principle as the official method of the American Leather Chemists Association.¹)

Digest overnight 30 grams of the fat-free leather, obtained under 5, in approximately 200 cc. of water. Transfer the leather and extract to a percolator. Continue the extraction by percolating with water at 50°C. Collect 2 liters of percolate, regulating the flow of water at such a rate that 2 liters will be collected in 3 hours. Dilute to volume at room temperature and mix thoroughly.

To the extract, prepared according to 6 or 7, add a few drops of toluol to prevent fermentation of sugars, and reserve for the determination of glucose, total solids, soluble solids, and nontannins.

GLUCOSE.

8

PREPARATION OF SOLUTION.

To 200 cc. of the leather extract, as prepared under 6 or 7, add 25 cc. of a saturated solution of normal lead acetate, mix thoroughly, and filter at once through a dry, plaited paper, returning the first portions of the filtrate to the filter until the filtrate becomes clear. Keep the containers and the funnel covered during these operations. Without waiting for the entire filtrate to run through add 10-12 grams of solid potassium oxalate, shake frequently during 15-20 minutes and filter through a dry, plaited paper returning the first runnings to the filter until the filtrate runs clear. Pipette 150 cc. of the last filtrate into a 600 cc. Erlenmeyer flask, add 5 cc. of concentrated hydrochloric acid and boil under a reflux condenser for 2 hours. Cool, neutralize with solid sodium carbonate, using a little phenolphthalein as indicator, transfer to a 200 cc. volumetric flask and complete to volume with water. Filter through a double filter, and return the first runnings until the filtrate becomes perfectly clear. Determine the dextrose in the filtrate immediately.

9 DETERMINATION.

Determine dextrose in 50 cc. of the solution, as prepared under 8, equivalent to 0.5 gram of leather, according to VIII, 25 and express the result as glucose.

10 TOTAL SOLIDS.

Determine as directed under V, 2.

11 SOLUBLE SOLIDS.

Determine as directed under V, 4.

12 NONTANNINS.

Determine as directed under V, 7.

13 SOLUBLE TANNIN.

The difference between the percentage of the soluble solids and the corrected nontannins is the percentage of tannin.

14 NITROGEN.

Determine as directed under I, 21.

15 HIDE SUBSTANCE.

Multiply the percentage of nitrogen by 5.62. The result will be the percentage of hide substance present.

16 COMBINED TANNIN.

Deduct the sum of the percentages of moisture, under 2, insoluble ash, under 4, soluble solids, under 11, and hide substance, under 15, from 100. The result will be the percentage of combined tannin.

BIBLIOGRAPHY.

¹ J. Am. Leather Chem. Assn., 1915, 10: 122.

VII. INSECTICIDES AND FUNGICIDES.

GENERAL METHOD.

1

PREPARATION OF SAMPLE.—TENTATIVE.

Mix thoroughly all samples before analysis. Make water-soluble arsenic determinations on samples as received without further pulverization or drying. In the case of lye, sodium cyanid or potassium cyanid, weigh large quantities in weighing bottles and analyze aliquots of the aqueous solutions.

PARIS GREEN.

2

MOISTURE.—TENTATIVE.

Dry 2 grams at 105°–110°C. for 5 hours and express the loss in weight as moisture.

TOTAL ARSENIC.—OFFICIAL.

(Arsenic, present as arsenate, is titrated as arsenious oxid.)

3

REAGENTS.

(a) *Starch indicator*.—Mix about 0.5 gram of finely powdered potato starch with cold water to a thin paste; pour into about 100 cc. of boiling water.

(b) *Standard arsenious oxid solution*.—Dissolve 2 grams of pure arsenious oxid in a beaker by boiling with about 150–200 cc. of water containing 10 cc. of concentrated sulphuric acid, cool, transfer to a 500 cc. graduated flask and dilute to the mark.

(c) *Standard iodine solution*.—Prepare an approximately N/20 solution as follows: Mix intimately 6.35 grams of pure iodine with twice its weight of pure potassium iodid. Dissolve in a small amount of water, filter and dilute the filtrate to 1 liter in a liter graduated flask. Standardize against (b) as follows: Pipette 50 cc. of the arsenious oxid into an Erlenmeyer flask, dilute to about 400 cc., neutralize with sodium bicarbonate, add 4–5 grams in excess, and add the standard iodine solution from a burette, shaking the flask continuously, until the yellow color disappears slowly from the solution, then add 5 cc. of the starch indicator and continue adding the iodine solution, drop by drop, until a permanent blue color is obtained. Calculate the value of the standard iodine solution in terms of *arsenious oxid* (As_2O_3) and *arsenic oxid* (As_2O_5). Occasionally restandardize the iodine against freshly prepared arsenious oxid solution.

4

APPARATUS.

The apparatus used is shown in Fig. 5. The distillation flask rests on a metal gauze which fits over a circular hole in a heavy sheet of asbestos board. The first 2 Erlenmeyer flasks are of 500 and 1000 cc. capacity and contain about 40 and 100 cc. of water, respectively. Both of these flasks should be placed in a pan and kept surrounded with cracked ice and water. The third flask, containing a small amount of water, is used as a trap.

5

DETERMINATION.

Weigh an amount of the sample equal to the arsenious oxid equivalent of 250 cc. of the standard iodine solution, and wash into the distillation flask by means of 100 cc. of concentrated hydrochloric acid (sp. gr. 1.19). Add 5 grams of cuprous chlorid (Cu_2Cl_2) and distil.

When the volume in the distillation flask is reduced to about 40 cc., add 50 cc. of concentrated hydrochloric acid by means of the dropping funnel and continue the distillation until 200 cc. of the acid distillate have passed over. Then wash down the condenser and all the connecting tubes carefully, transfer these washings and the contents of the 3 Erlenmeyer flasks to a liter graduated flask and dilute to the mark. Mix thoroughly, pipette 400 cc. into an Erlenmeyer flask and nearly neutralize with a saturated solution of sodium or potassium hydroxid, using a few drops of phenolphthalein as an indicator, keeping the solution well cooled.

Continue as directed under 3 (C) beginning with "neutralize with sodium bicarbonate." The number of cc. of iodine used in this titration represents directly the total per cent of arsenic in the sample expressed as arsenious oxid (As_2O_3).

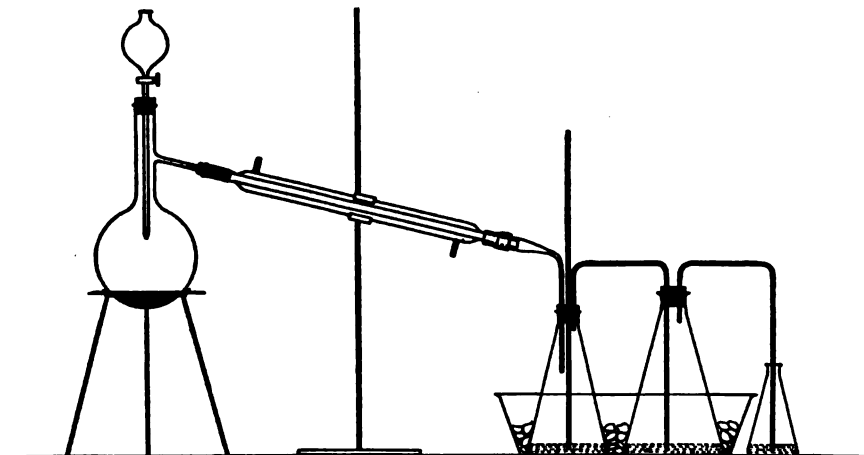


FIG. 5. APPARATUS FOR DISTILLATION OF ARSENIC CHLORID.

TOTAL ARSENIOS OXID.

(The following methods determine arsenic, and antimony if present, as the -ous oxids, As_2O_3 and Sb_2O_3 , respectively. Ferrous and cuprous salts vitiate the results.)

Method I.

C. C. Hedges Method,² Modified.³—Tentative.

6

REAGENTS.

The reagents and solutions used are described under 3.

7

DETERMINATION.

Weigh an amount of the sample equal to the arsenious oxid equivalent of 100 cc. of the standard iodine solution, wash into an Erlenmeyer flask with 10–15 cc. of dilute hydrochloric acid (1 to 1), followed by about 100 cc. of water, and heat on the steam-bath to complete solution, at a temperature not exceeding 60°C. Cool, neutralize

with sodium bicarbonate, add 4-5 grams in excess, and then sufficient 25% ammonium chlorid solution to dissolve the precipitated copper. Dilute somewhat and titrate as directed under 3 (C). A correction must be applied for the amount of iodine solution necessary to produce a blue color with starch in the presence of copper (using an equivalent weight of copper sulphate). The corrected number of cc. of the standard iodine solution used represents directly the per cent of arsenious oxid (As_2O_3) in the sample.

Method II.

8

C. M. Smith Method,¹ Modified.—Tentative.

Proceed as directed in 7, using dilute sulphuric acid (1 to 4) instead of dilute hydrochloric. The solution in this case may be heated to boiling.

SODIUM ACETATE-SOLUBLE ARSENIOUS OXID.—TENTATIVE.

9

REAGENTS.

(a) *Sodium acetate solution.*—Prepare a solution containing 12.5 grams of the crystallized salt ($CH_3COONa \cdot 3H_2O$) in each 25 cc.

The other reagents are described under 3.

10

DETERMINATION.

Place 1 gram of the sample in a 100 cc. flask and boil for 5 minutes with 25 cc. of the sodium acetate. Dilute to the mark, shake, and pass through a dry filter paper. Titrate an aliquot of this filtrate as directed under 3 (C). Calculate the amount of arsenious oxid (As_2O_3) present and express the result as per cent of sodium acetate-soluble arsenious oxid.

WATER-SOLUBLE ARSENIOUS OXID.—TENTATIVE.

11

REAGENTS.

Described under 3.

12

DETERMINATION.

To 1 gram of the sample in a liter Florence flask add 1 liter of recently boiled water which has been cooled to exactly 32°C. Stopper the flask and place in a water bath kept at 32°C. by means of a thermostat. Digest for 24 hours, shaking hourly for 8 hours during this period. Filter through a dry filter and titrate 250 cc. of the filtrate as directed under 3 (C). Correct for the amount of the standard iodine necessary to produce the same color, using the same reagents and volume. Calculate the amount of arsenious oxid (As_2O_3) present and express the result as per cent of water-soluble arsenious oxid.

TOTAL COPPER OXID.

13

Electrolytic Method.—Official.

Treat 2 grams of the sample in a beaker with 100 cc. of water and about 2 grams of sodium hydroxid and boil thoroughly until all the copper is precipitated as cuprous oxid. Filter, wash well with hot water, dissolve the precipitate in hot dilute nitric acid, cool, transfer to a 250 cc. graduated flask and dilute to the mark. (1) Use 50-100 cc. of this solution for the electrolytic determination of copper as directed under VIII, 33 and calculate to per cent cupric oxid; or, (2) Electrolyze the aliquot in a weighed 150 cc. platinum dish, using a rotating spiral anode and a current of about

3 amperes. After all the copper is deposited (requiring about 30 minutes), wash the deposit with water by siphoning, then rinse with alcohol, dry for a few minutes in an oven, weigh and calculate to per cent cupric oxid.

14

*Thiosulphate Method.*¹—Official.

Determine copper in another aliquot of the nitric acid solution of copper oxid, under 13, by titrating with N/20 thiosulphate solution, as directed under VIII, 29, and calculate to per cent cupric oxid.

LONDON PURPLE.

15

MOISTURE.—TENTATIVE.

Determined as directed under 2.

TOTAL ARSENIOUS OXID.¹—Official.

16

REAGENTS.

Described under 3.

17

DETERMINATION.

Dissolve 2 grams of the sample in a mixture of about 80 cc. of water and 20 cc. of concentrated hydrochloric acid at a temperature of 60°–70°C.; filter and wash until the combined filtrate and washings measure 250 cc. Treat 100 cc. of this solution with sodium bicarbonate in excess, transfer to a 500 cc. volumetric flask and make up to the mark, adding a few drops of ether to destroy the bubbles. Mix thoroughly and pass through a dry filter. Titrate 250 cc. of the filtrate as directed under 3 (C) and calculate the per cent of arsenious oxid.

TOTAL ARSENIC OXID.¹—Official.

18

REAGENTS.

The reagents and solutions used are described under 3.

19

DETERMINATION.

Boil, on a hot plate or over a low flame, 2 grams of the sample with 5 cc. of concentrated nitric acid and 20 cc. of concentrated sulphuric acid in a Kjeldahl digestion flask or a covered casserole. After 10–15 minutes add fuming nitric acid or powdered sodium nitrate, in small quantities at a time, until all organic matter is destroyed and the solution is practically colorless. Cool, add about 50 cc. of water (to decompose any nitro-sulphuric acid formed) and heat again until all nitric acid fumes are expelled. Cool, transfer to a 250 cc. volumetric flask, make up to the mark with water, mix thoroughly, and filter through a dry filter.

Transfer 50 cc. of this filtrate to a 400 cc. Erlenmeyer flask, dilute with water to 100 cc., add 1 gram of potassium iodid,² heat to boiling and evaporate to about 40 cc. (not less). Cool, dilute to 150–200 cc., and remove the excess of iodine with N/20 sodium thiosulphate. In case the solution is slightly colored from organic matter or from any cause other than free iodine, add the thiosulphate until it is nearly colorless, then a few drops of the starch indicator, and continue adding the thiosulphate slowly until the blue color just disappears. Continue at once as directed under 3 (C) beginning with "neutralize with sodium bicarbonate." Subtract from this reading the number of cc. of the standard iodine solution corresponding to the arsenious oxid obtained in 17. Calculate the per cent of arsenic oxid in the sample.

20

WATER-SOLUBLE ARSENIOS OXID.—TENTATIVE.

Proceed as directed under 12, slightly acidifying the aliquot employed with hydrochloric acid before adding the excess of sodium bicarbonate.

WATER-SOLUBLE ARSENIC OXID.—TENTATIVE.

21

REAGENTS.

The solutions and reagents used are described under 3.

22

DETERMINATION.

Transfer an aliquot, 250 cc., of the water extract, from 20, to a casserole, add 5 cc. of concentrated sulphuric acid, evaporate to a small volume and heat on a hot plate till white fumes of sulphuric acid appear. Cover the casserole and add 1-2 cc. of fuming nitric acid and again heat till the appearance of white fumes. Cool, add a little water and, in order to expel the last traces of nitric acid, once more evaporate till white fumes appear. Cool, dilute to about 100 cc. with water, add 1 gram of potassium iodid^s and sufficient sulphuric acid to make the total amount present about 5 cc. Boil until the volume is reduced to about 40 cc. Cool, dilute to about 200 cc., remove the excess iodine with N/20 sodium thiosulphate and proceed as directed under 3 (C) beginning with "neutralize with sodium bicarbonate." Correct for the amount of the standard iodine solution necessary to produce the same color, using the same reagents and volume. Subtract from the corrected titration reading the number of cc. of the standard iodine solution corresponding to the arsenious oxid, obtained in 20. Calculate the per cent of arsenic oxid present.

LEAD ARSENATE.

23

MOISTURE.—TENTATIVE.

(a) *Powder*.—Dry 2 grams to constant weight at 105°–110° C. and report the loss in weight as moisture.

(b) *Paste*.—Proceed as under (a), using 50 grams.

Grind the dry sample to a fine powder, mix well, transfer a small portion to a sample bottle and again dry for 1-2 hours at 105°–110°C., and use this anhydrous material for the determination of total lead oxid and total arsenic.

TOTAL LEAD OXID.

24

Method I.¹—Official.

Heat, on a hot plate, 0.6906 gram of the dry powdered sample with about 25 cc. of dilute nitric acid (1 to 4) in a 600 cc. beaker. If necessary, remove any insoluble residue by filtration. Dilute to at least 400 cc., heat nearly to boiling, add ammonium hydroxid to incipient precipitation, then dilute nitric acid (1 to 10) to redissolve the precipitate, adding 1-2 cc. in excess. Pipette into this solution, kept almost boiling, 50 cc. of a hot 10% potassium chromate solution, stirring constantly. Decant while hot through a weighed Gooch, previously heated at 140°–150°C., wash several times by decantation and then on the filter with boiling water until the washings are colorless. Dry the lead chromate at 140°–150°C. to constant weight. The weight of lead chromate multiplied by 100 gives the per cent of lead monoxid (PbO) in the dried sample.

The lead chromate precipitate may contain a small amount of lead arsenate which causes slightly high results. This error rarely amounts to more than 0.1–0.2%.

Method II.¹⁰—Tentative.

(Not applicable in the presence of calcium.)

25

REAGENT.

Acidified alcohol.—Mix water 100 parts; 95% alcohol 200 parts; and concentrated sulphuric acid 3 parts by volume.

26

DETERMINATION.

Heat, on a hot plate, 0.7360 gram of the dry powdered sample with about 25 cc. of dilute nitric acid (1 to 4) in a porcelain evaporating dish or casserole. Remove any insoluble residue by filtration. Add 3 cc. of concentrated sulphuric acid and evaporate on the hot plate to the appearance of white fumes. It is important that all nitric acid be expelled. Cool, add 50 cc. of water and about 100 cc. of 95% alcohol, let stand several hours (preferably over-night) and filter through a weighed Gooch crucible, previously washed with water, the acidified alcohol and 95% alcohol, and dried at 200°C. Wash the precipitate of lead sulphate in the crucible about 10 times with the acidified alcohol and then with 95% alcohol until free from sulphuric acid. Dry at 200°C. to constant weight, keeping the crucible covered to prevent loss by spattering. The weight of the lead sulphate multiplied by 100 gives the per cent of lead monoxid (PbO) in the dried sample.

TOTAL ARSENIC.

27

Method I.¹—Official.

Proceed as directed under 5, using an amount of the sample equal to the arsenic oxid equivalent of 500 cc. of the standard iodine solution and titrating a 200 cc. aliquot of the distillate. The number of cc. used of the standard iodine solution represents directly the total per cent of arsenic in the sample expressed as arsenic oxid (As_2O_3).

Method II.¹¹—Official.

(Not applicable in the presence of antimony.)

28

REAGENTS.

The reagents and solutions used are described under 3.

29

DETERMINATION.

Dissolve an amount of the powdered sample equal to the arsenic oxid equivalent of 400 cc. of the standard iodine solution, in dilute nitric acid in a porcelain casserole or evaporating dish. Add 5 cc. of concentrated sulphuric acid and heat on the hot plate to copious evolution of white fumes. Wash into a 200 cc. graduated flask with water, cool, make up to the mark and filter through a dry filter. Transfer 100 cc. of the filtrate to an Erlenmeyer flask and proceed as directed under 22, beginning with "add 1 gram of potassium iodide," to "Subtract from the corrected titration reading." The number of cc. of the standard iodine solution used, divided by 2, represents directly the per cent of total arsenic in the sample expressed as arsenic oxid (As_2O_3).

WATER-SOLUBLE ARSENIC OXID.—TENTATIVE.

30

REAGENTS.

The reagents and solutions used are described under 3.

31

DETERMINATION.

Treat 2 grams of the original sample, if in the form of a powder, or 4 grams, if a paste, as directed under 12 through "Filter through a dry filter."

Place 250–500 cc. of the *clear* filtrate in an Erlenmeyer flask, add 3 cc. of concentrated sulphuric acid and evaporate on a hot plate. When the volume is reduced to about 100 cc., proceed as directed under 22 to "Subtract from the corrected titration reading." Calculate and report as per cent of water-soluble arsenic oxid (As_2O_3).

CALCIUM ARSENATE.

32

TOTAL ARSENIC.—OFFICIAL.

Proceed as directed under 5, using an amount of the powdered sample equal to the arsenic oxid equivalent of 250 cc. of the standard iodine solution.

The number of cc. of the standard iodine solution used represents directly the total per cent of arsenic in the sample expressed as arsenic oxid (As_2O_3).

ZINC ARSENITE.

33

TOTAL ARSENIC.—OFFICIAL.

Proceed as directed under 5, using an amount of the powdered sample equal to the arsenious oxid equivalent of 500 cc. of the standard iodine solution and titrating a 200 cc. aliquot of the distillate. The number of cc. of the standard iodine solution used represents directly the per cent of total arsenic in the sample expressed as arsenious oxid (As_2O_3).

34

TOTAL ARSENIOUS OXID.—TENTATIVE.

Proceed as directed under 7 or 8.

COPPER CARBONATE.

35

COPPER OXID.—OFFICIAL.

Dissolve a weighed quantity of the substance in dilute nitric acid and determine copper as directed under 13 or 14.

BORDEAUX MIXTURE.

36

MOISTURE.—OFFICIAL.

(a) *Powder*.—Dry 2 grams to constant weight at 105°–110°C. and express the loss in weight as moisture.

(b) *Paste*.—Heat about 100 grams in an oven at 90–100°C. until dry enough to powder readily, and note the loss in weight. Powder this partially dried sample, and determine the remaining moisture in 2 grams as under (a). Determine carbon dioxid, as directed under 38, both in the original paste and in this partially dried sample. Calculate the total moisture by the following formula:

$M = a + (100-a)(b + c) - d$ in which

M = per cent total moisture in original paste;

a = per cent loss in weight of original paste during first drying;

b = per cent loss in weight of partially dried paste during second drying;

c = per cent carbon dioxid remaining in partially dried paste after first drying;

d = per cent total carbon dioxid in original paste.

CARBON DIOXID.¹²—OFFICIAL.

37

APPARATUS.

This consists of a 200 cc. Erlenmeyer flask closed with a 2-holed stopper; one of these holes is fitted with a dropping funnel the stem of which extends almost to the bottom of the flask; the outlet of a condenser, which is inclined upward at an angle of 30° from the horizontal, passes downward through the other hole. The upper end of the condenser is connected with a calcium chlorid tube which in turn is connected with a double U-tube filled in the middle with pumice fragments, previously saturated with copper sulphate solution and subsequently dehydrated, and with calcium chlorid at either end. Then follow 2 weighed U-tubes for absorbing the carbon dioxid, the first filled with porous soda-lime, and the second, one third with soda-lime and two thirds with calcium chlorid, the latter reagent being placed at the exit end of the train. A Geissler bulb, partly filled with sulphuric acid, is attached to the last U-tube to show the rate of gas flow. An aspirator is connected with the Geissler bulb to draw air through the apparatus. An absorption tower filled with soda-lime is connected with the mouth of the dropping funnel to remove carbon dioxid from the air entering the apparatus.

38

DETERMINATION.

Weigh 2 grams of the powder or 10 grams of the paste into the Erlenmeyer flask, add about 20 cc. of water, attach the flask to the apparatus omitting the 2 weighed U-tubes, and draw carbon dioxid-free air through the apparatus until the original air is displaced. Then attach the weighed U-tubes in the position as described in 37, close the stop-cock of the dropping funnel, fill half full with dilute hydrochloric acid (1 to 1), reconnect with the soda-lime tower, and allow the acid to flow into the Erlenmeyer flask, slowly if there is much carbon dioxid, rapidly if there is little. When effervescence diminishes, place a low Bunsen flame under the flask and start a flow of water through the condenser, a slow current of air being allowed to flow through the apparatus at the same time. Maintain a steady but quiet ebullition, and a slow air current through the apparatus. Boil for a few minutes after the water has begun to condense in the condenser, then remove the flame and continue the aspiration of air at the rate of about 2 bubbles per second until the apparatus is cool. Disconnect the tared absorption tubes, cool in the balance case and weigh. The increase in weight is due to carbon dioxid.

COPPER.

39

Electrolytic Method.—Official.

Dissolve 2 grams of the dry powdered sample in 20 cc. of water and 5 cc. of concentrated nitric acid, dilute to 100 cc., wash into a weighed 150 cc. platinum dish, and electrolyze, using a rotating spiral anode and a current of about 3 amperes. After all the copper is deposited (requiring about 30 minutes), wash the deposit with water by siphoning, then rinse with alcohol, dry for a few minutes in an oven, and weigh. Calculate the per cent of copper in the sample.

40

Thiosulphate Method.—Official.

Dissolve 2 grams of the dry powdered sample in about 50 cc. of 10% nitric acid, add ammonium hydroxid solution in excess and heat; then, without removing the precipitate which is formed, boil off the excess of ammonia, add 5-10 cc. of acetic acid, cool, add 10 cc. of 30% potassium iodid solution, and titrate as directed under VIII, 29.

BORDEAUX MIXTURE WITH PARIS GREEN.

41

MOISTURE.—OFFICIAL.

Proceed as directed under 36.

42

CARBON DIOXID.—OFFICIAL.

Proceed as directed under 38.

COPPER.

43

Method I.—Tentative.

Dissolve 2 grams of the dry powdered sample in a few cc. of strong nitric acid, add 25 cc. of a 3% solution of hydrogen peroxid and warm for 5–10 minutes. Make slightly alkaline with ammonium hydroxid and then slightly acid again with dilute nitric acid. Transfer to a weighed 150 cc. platinum dish, add 15–20 cc. of hydrogen peroxid, dilute to 100 cc. and electrolyze, using a rotating spiral anode and a current not exceeding 2 amperes. After the electrolysis has proceeded for about 20 minutes, add to the electrolyte 0.5 gram of ferric sulphate dissolved in a few cc. of water together with a drop or two of nitric acid. After all the copper is deposited, wash the deposit with water by siphoning, then rinse with alcohol, dry for a few minutes in an oven, weigh and calculate the per cent of copper. (Do not pass the current for more than 5–10 minutes after all the copper has been deposited without adding more ferric sulphate solution.)

44

Method II.—Tentative.

Treat 1 gram of the dry powdered sample with 20 cc. of water and 5–6 cc. of concentrated nitric acid, heat to boiling, cool, and add a slight excess of concentrated ammonium hydroxid. Wash the solution and precipitate into a weighed platinum dish of about 150 cc. capacity, and electrolyze, using a rotating anode and a current of about 4 amperes and 3–4 volts for about 90 minutes (or until all the copper is deposited). Wash the deposit by siphoning until the deposit is clean, being careful not to use too much wash water. Dissolve the copper in 5 cc. of concentrated nitric acid, dilute to 100 cc. and electrolyze as before, except that all the copper will be deposited in 30 minutes. Wash the deposit with water by siphoning, then rinse with alcohol, dry for a minute or so in an oven, weigh and calculate the per cent of copper.

45

TOTAL ARSENIC.—OFFICIAL.

Proceed as directed under 5, using an amount of the dry powdered sample equal to the arsenious oxid equivalent of 500 cc. of the standard iodine solution. The number of cc. of the standard iodine solution used, divided by 2, represents directly the per cent of total arsenic in the sample expressed as arsenious oxid (As_2O_3).

TOTAL ARSENIUS OXID.

46

Method I.—Tentative.

Proceed as directed under 7, using an amount of the dry, powdered sample equal to the arsenious oxid equivalent of 200 cc. of the standard iodine solution. Before titrating, all the copper must be in solution. The corrected number of cc. of the standard iodine solution used, divided by 2, represents directly the per cent of total arsenious oxid (As_2O_3) in the sample.

47

Method II.—Tentative.

Proceed as directed under 8.

48

WATER-SOLUBLE ARSENIOUS OXID.—TENTATIVE.

Proceed as directed under 20, using 2 grams of the sample.

BORDEAUX MIXTURE WITH LEAD ARSENATE.

49

MOISTURE.—OFFICIAL.

Proceed as directed under 36.

50

CARBON DIOXID.—OFFICIAL.

Proceed as directed under 38.

51

COPPER.—TENTATIVE.

Proceed as directed under 44.

52

LEAD OXID.—TENTATIVE.

Dissolve the lead peroxid (which will contain a little arsenic) from the anodes used in the copper electrolysis, under 51, by means of dilute nitric acid and a little hydrogen peroxid, and add to this solution the washings from both electrolyses of copper. Add ammonium chlorid to dissolve any lead sulphate which may have precipitated out and make the solution up to 1 liter. Concentrate a 500 cc. aliquot of this solution to about 300 cc. (all hydrogen peroxid must be expelled from the solution), transfer to a 400 cc. beaker and precipitate the lead as lead chromate as directed under 24.

53

TOTAL ARSENIC.—OFFICIAL.

Proceed as directed under 5, using an amount of the dry, powdered sample equal to the arsenic oxid equivalent of 500 cc. of the standard iodine solution. The number of cc. of the standard iodine solution used, divided by 2, represents directly the per cent of total arsenic in the sample expressed as arsenic oxid (As_2O_3).

54

WATER-SOLUBLE ARSENIC OXID.—TENTATIVE.

Proceed as directed under 31.

SODIUM AND POTASSIUM CYANIDS.

55

CYANOGEN.¹²—OFFICIAL.

Weigh about 10 grams of the sample in a weighing bottle, dissolve in water, and make up to volume in a liter graduated flask. To a 50 cc. aliquot add N/20 silver nitrate, drop by drop, stirring constantly, until 1 drop produces a permanent turbidity. In calculating the results, 1 equivalent of silver is equal to 2 equivalents of cyanogen, according to the following equation:



Reserve the titrated solution for the determination of chlorin under 56.

56

CHLORIN.¹⁴—OFFICIAL.

After completion of the titration for cyanogen, as directed under 55, add a few cc. of 10% potassium chromate solution as indicator and titrate with N/20 silver nitrate until the appearance of the red-brown color of silver chromate.

The first titration with silver nitrate represents the cyanogen present according to the equation above. The second titration represents the cyanogen and chlorin according to the following equation: $\text{NaCNAgCN} + \text{NaCl} + 2\text{AgNO}_3 = 2\text{NaNO}_3 + 2\text{AgCN} + \text{AgCl}$. Therefore the second minus the first reading represents the chlorin present in terms of silver nitrate.

SOAP.

MOISTURE.

57

*Modified Method of Benedickt and Lewkowitsch.*¹⁵—Tentative.

Weigh about 5 grams of the sample in a tared, 100 cc. beaker, in which is previously placed a $\frac{1}{2}$ inch layer of recently ignited, dry sand, and a small glass rod; if the soap is hard, cut off the soap in very thin strips. Add 25 cc. of alcohol, or more if necessary, and dissolve on the water bath, stirring constantly. Evaporate the alcohol, heat in an oven at 110°C. until the soap is nearly dry, and weigh, then dry again for 30 minutes and weigh. Continue this alternate drying and weighing until the weight changes only a few milligrams during the course of 30 minutes' drying.

58

POTASSIUM AND SODIUM.¹⁶—TENTATIVE.

Dissolve about 5 grams of the soap in water; decompose with hydrochloric acid, filter off the water and wash the fat with cold water. Determine both potassium and sodium in the filtrate as directed under II, 21.

SODA LYE.

59

CARBONATE AND HYDROXID.¹⁷—OFFICIAL.

Weigh about 10 grams of the sample from the weighing bottle, dissolve in carbon dioxid-free water and make up to a definite volume. Titrate an aliquot of this solution with N/2 hydrochloric acid, using methyl orange as an indicator, and note the total alkalinity thus found. Transfer an equal aliquot to a graduated flask and add enough barium chlorid solution to precipitate all the carbonate, avoiding any unnecessary excess. Dilute to the mark with carbon dioxid-free water, stopper, shake, and set aside. When the liquid becomes clear, pipette off one half and titrate with N/2 hydrochloric acid, using phenolphthalein as an indicator. The number of cc. of N/2 acid, required for this titration, multiplied by 2 gives the number of cc. of N/2 acid required to neutralize the sodium hydroxid present in the original aliquot. The difference between this figure and the number of cc. of N/2 hydrochloric acid required for the total alkalinity represents the number of cc. of N/2 acid required to neutralize the sodium carbonate present in the aliquot. Calculate the percentages of sodium carbonate and hydroxid present in the sample.

TOBACCO AND TOBACCO EXTRACT.

NICOTIN.

Kissling Method.—Official.

60

REAGENTS.

(a) *Alcoholic sodium hydroxid solution.*—Dissolve 6 grams of sodium hydroxid in 40 cc. of water and 60 cc. of 90% alcohol.

- (b) 0.4% sodium hydroxid solution.
- (c) N/10 sulphuric acid.—One cc. is equivalent to 16.22 mg. of nicotin.
- (d) Phenacetolin solution.—Prepare a 0.5% alcoholic solution.
- (e) Cochineal solution.—Prepare as directed under I, 16 (k).

61

DETERMINATION.

Weigh 5-6 grams of tobacco extract, or 20 grams of finely powdered tobacco which has been previously dried at 60°C. if necessary, into a small beaker. Add 10 cc. of the alcoholic sodium hydroxid and follow, in the case of tobacco extract, with enough pure powdered calcium carbonate to form a moist but not lumpy mass. Mix thoroughly, transfer to a Soxhlet extractor and exhaust for about 5 hours with ether. Evaporate the ether at a low temperature, and take up the residue with 50 cc. of the 0.4% sodium hydroxid solution. Transfer this residue by means of water to a 500 cc. Kjeldahl flask, and distil with steam, passing the distillate through a condenser cooled by a rapidly flowing current of water. Use a 3-bend outflow tube, and, to prevent bumping and frothing, add a few pieces of pumice, and a small piece of paraffin. Distil till all the nicotin has passed over, the distillate usually varying from 400-500 cc. When completed, only about 15 cc. of the liquid should remain in the flask. Titrate the distillate with N/10 sulphuric acid, using the phenacetolin or cochineal solution as indicator.

Silicotungstic Acid Method.^{1a}—Official.

62

REAGENTS.

- (a) *Silicotungstic acid solution*.—Prepare a 12% solution of the silicotungstic acid having the following formula: $4\text{H}_2\text{O} \cdot \text{SiO}_2 \cdot 12\text{WO}_3 \cdot 22\text{H}_2\text{O}$.
- (b) *Sodium or potassium hydroxid solution (1 to 2)*.
- (c) *Dilute hydrochloric acid (1 to 4)*.

63

DETERMINATION.

Weigh such an amount of the preparation as will contain preferably between 0.1 and 1.0 gram of nicotin (if the sample contains very little nicotin, about 0.1%, do not increase the amount to the point where it interferes with the distillation); wash with water into a 500 cc. round-bottomed distillation flask; add a little paraffin to prevent frothing, a few small pieces of pumice and a slight excess of the sodium or potassium hydroxid, using phenolphthalein as an indicator. Distil rapidly in a current of steam through a well-cooled condenser, connected by means of an adapter with a suitable flask containing 10 cc. of the dilute hydrochloric acid. When distillation is well under way, heat the distillation flask to reduce the volume of the liquid as far as practicable without bumping or undue separation of insoluble matter. Distil until a few cc. of the distillate show no cloud or opalescence when treated with a drop of the silicotungstic acid and a drop of the dilute hydrochloric acid. Confirm the alkalinity of the residue in the distillation flask with phenolphthalein solution. Make up the distillate, which may amount to 1000-1500 cc., to a convenient volume (the solution may be concentrated on the steam bath without loss of nicotin), mix well and pass through a large dry filter if not clear. Test a portion with methyl orange to assure its acidity. Pipette an aliquot, containing about 0.1 gram of nicotin, into a beaker (if the samples contain very small amounts of nicotin, an aliquot containing as little as 0.01 gram of nicotin may be used), add to each 100 cc. of liquid 3 cc. of the dilute hydrochloric acid, or more if the necessity is indicated by the test with methyl orange, and add 1 cc. of the silicotungstic acid for each 0.01 gram of nico-

tin supposed to be present. Stir thoroughly and let stand overnight. Before filtering, stir the precipitate to see that it settles quickly and is in crystalline form; then filter on an ashless filter paper, and wash with cold dilute hydrochloric acid (1 to 1000). Transfer the paper and precipitate to a weighed platinum crucible, dry carefully, and ignite until all carbon is destroyed. Finally heat over a Teclu or Meker burner for not more than 10 minutes. The weight of the residue multiplied by 0.114 gives the weight of nicotin present in the aliquot.

FORMALDEHYDE SOLUTIONS.

FORMALDEHYDE.

*Hydrogen Peroxid Method.*¹²—Official.

64

REAGENTS.

- (a) *N/1 sulphuric acid.*
- (b) *N/1 sodium hydroxid.*—One cc. is equivalent to 30.02 mg. of formaldehyde.
- (c) *Hydrogen peroxid.*—An approximately 3% solution. If the hydrogen peroxid solution is acid, neutralize with (b), using litmus solution as indicator.
- (d) *Litmus solution.*—A solution of purified litmus.

65

DETERMINATION.

Measure 50 cc. of *N/1* sodium hydroxid into a 500 cc. Erlenmeyer flask and add 50 cc. of the hydrogen peroxid. Then add 3 grams of the formaldehyde solution under examination, allowing the point of the pipette to reach nearly to the liquid in the flask. Place a funnel in the neck of the flask and heat on the steam bath for 5 minutes, shaking occasionally. Remove from the steam bath, wash the funnel with water, cool the flask to about room temperature, and titrate with *N/1* acid, using the litmus solution as indicator. It is necessary to cool the flask before titration with the acid to get a sharp end point with the litmus. Calculate the per cent of formaldehyde.

*Cyanid Method.*¹²—Official.

66

REAGENTS.

- (a) *N/10 silver nitrate.*
- (b) *N/10 ammonium sulphocyanate.*
- (c) *Potassium cyanid solution.*—Dissolve 3.1 grams of potassium cyanid in 500 cc. of water.
- (d) *50% nitric acid.*

67

DETERMINATION.

Treat 15 cc. of the *N/10* silver nitrate with 6 drops of the 50% nitric acid in a 50 cc. volumetric flask; add 10 cc. of the potassium cyanid solution, dilute to the mark, shake well, filter through a dry filter and titrate 25 cc. of the filtrate with *N/10* ammonium sulphocyanate as directed under III, 15. Acidify another 15 cc. portion of the *N/10* silver nitrate with 6 drops of the 50% nitric acid and treat with 10 cc. of the potassium cyanid solution to which has been added a measured quantity (the weight of which must be calculated from the specific gravity) of the formaldehyde solution containing not over 2.5 grams of a 1% solution or the equivalent. Make up to 50 cc., filter and titrate a 25 cc. aliquot with the *N/10* ammonium sulphocyanate for the excess of silver as before. The difference between the number of cc. of *N/10* ammonium sulphocyanate used in these 2 titrations, multiplied

by 2, gives the number of cc. of N/10 ammonium sulphocyanate corresponding to the potassium cyanid used by the formaldehyde. Calculate the per cent of formaldehyde present (1 cc. of N/10 ammonium sulphocyanate is equivalent to 3 mg. of formaldehyde (HCHO)).

LIME-SULPHUR SOLUTIONS.²¹

TOTAL SULPHUR.—OFFICIAL.

68

PREPARATION OF SOLUTION.

Weigh 10 grams of the solution and dilute to the mark in a 250 cc. graduated flask with recently boiled and cooled water.

69

DETERMINATION.

Transfer a 10 cc. aliquot to a 400 cc. beaker, add about 3 grams of sodium peroxid, cover immediately with a watch glass and warm on the steam bath, with frequent shaking, until all the sulphur is oxidized to sulphate, adding more sodium peroxid if necessary. Dilute, acidify with hydrochloric acid, evaporate to dryness, treat with water acidified with hydrochloric acid, boil, and filter to remove silica, if present. Dilute the filtrate to 300 cc., add 50 cc. of concentrated hydrochloric acid,²² heat to boiling, and precipitate with 10% barium chlorid solution slowly and stirring constantly. (The rate is best regulated by attaching a suitable capillary tip to the burette containing the barium chlorid solution.) Evaporate to dryness on the steam bath, take up with hot water, filter through a quantitative filter paper, wash until free from chlorin, ignite and heat to constant weight over a Bunsen burner. Calculate the sulphur from the weight of barium sulphate. Previous to use test the reagents for sulphur and, if present, make corrections accordingly.

SULPHID SULPHUR.—OFFICIAL.

70

REAGENT.

Ammoniacal zinc solution.—Dissolve 50 grams of pure zinc chlorid in water, add ammonium hydroxid in sufficient quantity to redissolve the precipitate first formed, then add 50 grams of ammonium chlorid²³ and dilute to 1 liter.

71

DETERMINATION.

Dilute 10 cc. of the solution, prepared as directed under 68, to about 100 cc. and add the ammoniacal zinc solution until the sulphid is all precipitated, indicated by the addition of a drop of the clear solution to a few drops of nickel sulphate solution. Filter immediately, wash the precipitate thoroughly with cold water and transfer it and the filter paper to a beaker. Cover with water, disintegrate with a glass rod and add about 3 grams of sodium peroxid, keeping the beaker well covered with a watch glass. Warm on the steam bath with frequent shaking until all the sulphur is oxidized to sulphate, adding more sodium peroxid if necessary. Make slightly acid with hydrochloric acid, filter to remove shreds of filter paper, wash thoroughly with hot water, and determine the sulphur in the filtrate exactly as under 69.

72

THIOSULPHATE SULPHUR.—OFFICIAL.

Dilute 50 cc. of the solution, prepared as under 68, to about 100 cc. in a 200 cc. graduated flask. Add a slight excess of the ammoniacal zinc chlorid and dilute to the mark. Shake thoroughly and filter through a dry filter. To 100 cc. of the filtrate add a few drops of methyl orange and exactly neutralize with N/10 hydrochloric

acid. Titrate this neutral solution with approximately N/20 iodine, 3 (C), using a few drops of starch solution as indicator. From the number of cc. of iodine solution used, calculate the thiosulphate sulphur present.

73

SULPHATE SULPHUR.—OFFICIAL.

To the solution from the determination in 72, add 2 or 3 drops of hydrochloric acid, precipitate in the cold with 10% barium chlorid solution, allow to stand overnight, filter, calculate the sulphur from the weight of barium sulphate and report as sulphate sulphur.

74

TOTAL LIME.—OFFICIAL.

To 25 cc. of the solution, prepared as under 68, add 10 cc. of concentrated hydrochloric acid, evaporate to dryness on the steam bath, treat with water and a little hydrochloric acid, warm until all the calcium chlorid is dissolved, and filter from sulphur and any silica that may be present. Oxidize the filtrate by boiling with a little concentrated nitric acid, make ammoniacal, filter from iron and aluminium if present, heat to boiling and precipitate the calcium with ammonium oxalate solution. Filter, wash and ignite over a blast lamp to constant weight; weigh the residue as calcium oxid.

BIBLIOGRAPHY.

- ¹ J. Ind. Eng. Chem., 1916, 8: 327.
- ² Ibid., 1909, 1: 208.
- ³ J. Assoc. Official Agr. Chemists, 1915, 1: 436, 446.
- ⁴ J. Am. Chem. Soc., 1901, 23: 115.
- ⁵ Ibid., 1902, 24: 1082.
- ⁶ Ibid., 1900, 22: 802.
- ⁷ U. S. Bur. Chem. Bull. 122, p. 106.
- ⁸ Am. J. Sci., 1890, 3rd ser., 40: 66.
- ⁹ U. S. Bur. Chem. Bull. 137, p. 40.
- ¹⁰ Ibid., 105, p. 166.
- ¹¹ Ibid., p. 167.
- ¹² Fresenius. Quantitative Chemical Analysis. Revised and amplified translation of the 6th German ed., 2: 1180; U. S. Geol. Surv. Bull. 422, p. 179.
- ¹³ Sutton. Volumetric Analysis. 10th ed., 1911, p. 207.
- ¹⁴ Ibid., 9th ed., rev., p. 201.
- ¹⁵ Lewkowitsch. Chemical Technology and Analysis of Oils, Fats and Waxes. 5th ed., 1915, 3: 348.
- ¹⁶ Ibid., 346.
- ¹⁷ Sutton. Volumetric Analysis. 10th ed., 1911, p. 61.
- ¹⁸ U. S. Bur. Animal Industry, Bull. 133.
- ¹⁹ Ber., 1898, 31: 2979; J. Am. Chem. Soc., 1905, 27: 1183; U. S. Bur. Chem. Bull. 99, p. 30; 132, p. 49; 137, p. 47.
- ²⁰ Z. anal. Chem., 1897, 36: 18; U. S. Bur. Chem. Bull. 132, p. 49.
- ²¹ J. Assoc. Official Agr. Chemists, 1915, 1: 76.
- ²² J. Am. Chem. Soc., 1911, 33: 844.
- ²³ J. Soc. Chem. Ind., 1912, 31: 369.

VIII. FOODS AND FEEDING STUFFS.

1

PREPARATION OF SAMPLE.—OFFICIAL.

Grind the sample so that it will pass through a sieve having circular openings $\frac{1}{16}$ inch (1 mm.) in diameter. If the sample can not be ground, reduce it to as fine a state as possible.

MOISTURE.

2

Direct Drying.—Official.

Dry a quantity of the substance, representing about 2 grams of dry material, in a current of dry hydrogen or in vacuo at the temperature of boiling water to constant weight (approximately 5 hours). If the substance be held in a glass vessel, the latter should not be in contact with the boiling water.

3

Drying in Vacuo without Heat.—Tentative.

Mix the sample thoroughly and weigh by difference 2–5 gram portions from a stoppered weighing bottle into tared, covered crucibles. Where subsequent fat determinations are to be made, fat extraction cones may be used. Substances that dry down to horn-like material should be mixed with fat-free cotton or other suitable material (previously tared with the container). Place 200 cc. of fresh concentrated sulphuric acid in a strong, tight 6 inch vacuum desiccator. Put triplicate samples in separate desiccators, and exhaust by means of a vacuum pump. If a pump is not available, place 10 cc. of ether in a small beaker in the desiccator, and exhaust with a water filter pump.

Between the pump and the desiccator interpose an empty bottle, next to the desiccator, and a bottle of water. Draw the air from the desiccator through the water and turn the desiccator stop-cock at just the instant when the water begins to rise in the tube leading from the empty bottle.

Gently rotate the desiccator 4 or 5 times during the first 12 hours to mix the sulphuric acid with the water which has collected as an upper layer. At the end of 24 hours open the desiccator, forcing the incoming air to bubble through concentrated sulphuric acid, and make the first weighing. After weighing place in a desiccator containing fresh concentrated sulphuric acid and exhaust as before. Rotate the desiccator several times during the interval and weigh again after a suitable period of drying. Repeat this process of drying in vacuo over sulphuric acid until the weight is constant.

4

ASH.—OFFICIAL.

Char a quantity of the substance, representing about 2 grams of the dry material, and burn until free from carbon at a low heat, not to exceed dull redness. If a carbon-free ash can not be obtained in this manner, exhaust the charred mass with hot water, collect the insoluble residue on a filter, burn till the ash is white or nearly so, and then add the filtrate to the ash and evaporate to dryness. Heat to low redness till the ash is white or grayish white and weigh.

5

CRUDE PROTEIN.—OFFICIAL.

Determine nitrogen as directed under I, 18, 21, or 23, and multiply the result by 6.25.

ALBUMINOID NITROGEN.—OFFICIAL.

6

REAGENT.

Stutzer's reagent.—Prepare cupric hydroxid as follows: Dissolve 100 grams of pure copper sulphate in 5 liters of water, add 2.5 cc. of glycerol, and then dilute sodium hydroxid solution until the liquid is just alkaline; filter, rub the precipitate up with water containing 5 cc. of glycerol per liter, and wash by decantation or filtration until the washings are no longer alkaline. Rub the precipitate up again in a mortar with water containing 10% of glycerol, thus preparing a uniform gelatinous mass that can be measured with a pipette. Determine the quantity of copper hydroxid per cc. of this mixture.

7

DETERMINATION.

Place 0.7 gram of the substance in a beaker, add 100 cc. of water, and heat to boiling; or, in case of substances rich in starch, heat on the water bath for 10 minutes; add a quantity of the Stutzer's reagent containing about 0.5 gram of the hydroxid; stir thoroughly, filter when cold, wash with cold water, and, without removing the precipitate from the filter, determine the nitrogen according to I, 18, 21 or 23, adding sufficient potassium sulphid solution to completely precipitate all of the copper and mercury. The filter paper used must be practically free from nitrogen. If the material (such as seeds, seed residue, or oil cake) is rich in alkaline phosphates, add, to decompose the alkaline phosphates, 1-2 cc. of a concentrated potash or soda alum solution, free from ammonia, then the copper hydroxid, and mix well by stirring. If this is not done, copper phosphate and free alkali may be formed, and the protein-copper precipitate partially dissolved in the alkaline liquid.

8

AMIDO NITROGEN.—OFFICIAL.

Subtract the amount of albuminoid nitrogen from the amount of total nitrogen to obtain the amido nitrogen.

CRUDE FAT OR ETHER EXTRACT.

Direct Method.—Official.

9

REAGENT.

Anhydrous ether.—Wash any of the commercial brands of ether with 2 or 3 successive portions of water, add solid sodium or potassium hydroxid, and let stand until most of the water has been abstracted from the ether. Decant into a dry bottle, add small pieces of carefully cleaned metallic sodium, and let stand until there is no further evolution of hydrogen gas. Keep the ether, thus dehydrated, over metallic sodium in lightly stoppered bottles.

10

DETERMINATION.

Large quantities of soluble carbohydrates may interfere with the complete extraction of the fat. In such cases extract with water before proceeding with the determination. Extract about 2 grams of material, dried as under 2 or 3, with the anhydrous ether for 16 hours. Dry the extract at the temperature of boiling water for 30 minutes, cool in a desiccator, and weigh; continue, at 30 minutes intervals, this alternate drying and weighing to constant weight. For most feeds a period of 1-1½ hours is required.

11

Indirect Method.—Official.

Determine the moisture, as directed in 2 or 3, then extract the dried substance for 16 hours as directed under 10, dry again and regard the loss of weight as ether extract.

SUCROSE.

OPTICAL METHODS.

12

GENERAL DIRECTIONS FOR RAW SUGARS.—TENTATIVE.

(Rules¹ of the International Commission for Unifying Methods of Sugar Analysis.)

"In general all polarizations are to be made at 20°C."

"The verification of the saccharimeter must also be made at 20°C. For instruments using the Ventske scale 26 grams of pure dry sucrose, weighed in air with brass weights, dissolved in 100 metric cc. at 20°C. and polarized in a room, the temperature of which is also 20°C., must give a saccharimeter reading of exactly 100.00. The temperature of the sugar solution during polarization must be kept constant at 20°C."

"For countries where the mean temperature is higher than 20°C., saccharimeters may be adjusted at 30°C. or any other suitable temperature, under the conditions specified above, provided that the sugar solution be made up to volume and polarized at this same temperature."

"In effecting the polarization of substances containing sugar employ only half-shade instruments." The saccharimeter used can be either single or double wedge and should be a half-shadow instrument with either double or triple field.

"During the observation keep the apparatus in a fixed position and so far removed from the source of light that the polarizing Nicol is not warmed."

"As sources of light employ lamps which give a strong illumination such as triple gas burner with metallic cylinder, lens and reflector; gas lamps with Auer (Welsbach) burner; electric lamp; petroleum duplex lamp; sodium light." Whenever there is any irregularity in the sources of light such as that due to the convolutions of the filament in the case of electric light or to the meshes of the gauze in the case of the Welsbach light, place a thin ground-glass plate between the source of light and the polariscope so as to render the illumination uniform.

"Before and after each set of observations the chemist must satisfy himself of the correct adjustment of his saccharimeter by means of standardized quartz plates. He must also previously satisfy himself of the accuracy of his weights, polarization flasks, observation tubes and cover-glasses. (Scratched cover-glasses must not be used.) Make several readings and take the mean thereof, but no one reading may be neglected." Such plates are standardized to read to the second decimal point and by their use a quick and at the same time accurate test can be made. In using such plates for testing saccharimeters, it is necessary that the instrument, as well as the plate, be at 20°C. before making a reading. Different points of the scale, preferably 20°, 50°, 80°, and 100°, (sugar scale) should be tested against the plates.

"In making a polarization use the whole normal weight for 100 cc. or a multiple thereof for any corresponding volume."

"As clarifying and decolorizing agents use either basic acetate of lead, alumina cream, or concentrated solution of alum. Boneblack and decolorizing powders are to be excluded." Whenever reducing sugars are determined in the solution for polarizing, use only neutral lead acetate for clarification as basic lead acetate causes precipitation of some of the reducing sugars. In addition to these clarifying agents,

neutral lead acetate and basic lead nitrate (Herles' solution) have been made official by the Association.

"After bringing the solution exactly to the mark at the proper temperature, and after wiping out the neck of the flask with filter paper, pour all of the well-shaken clarified sugar solution on a rapidly acting filter. Reject the first portions of the filtrate, and use the rest, which must be perfectly clear, for polarization." It is advisable to reject the first 20 cc. that run through, then cover the funnel with a watch glass and use the remainder for polarization. In no case should the whole solution or any part be returned to the filter. If cloudy after the 20 cc. have been rejected, begin a new determination.

"Whenever white light is used in polarimetric determinations, the same must be filtered through a solution of potassium dichromate of such a concentration that the percentage content of the solution multiplied by the length of the column of the solution in centimeters is equal to nine." This concentration must be doubled in reading carbohydrate materials of high rotation dispersion, such as commercial glucose, etc.

13

PREPARATION AND USE OF CLARIFYING REAGENTS.—TENTATIVE.

(a) *Basic lead acetate solution*.—Boil 430 grams of neutral lead acetate, 130 grams of litharge, and 1 liter of water for 30 minutes. Allow the mixture to cool and settle and dilute the supernatant liquid to a specific gravity of 1.25 with recently boiled water. Solid basic lead acetate may be substituted for the normal salt and litharge in the preparation of the solution.

(b) *Alumina cream*.—Prepare a cold saturated solution of alum in water. Add ammonium hydroxid with constant stirring until the solution is alkaline to litmus, allow the precipitate to settle and wash by decantation with water until the wash water gives only a slight test for sulphates with barium chlorid solution. Pour off the excess of water and store the residual cream in a stoppered bottle.

(c) *Dry basic lead acetate (Horne method)*.—This clarifying agent is obtained as a dry powdered salt and should contain 72.8% of lead, which corresponds to a composition of $3\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{PbO}$. Dissolve the normal or half-normal weight of the sugar solution in a flask with water and complete the volume. Add a small quantity of the dry salt and shake, then add more and shake again, repeating until completely precipitated but avoiding any excess. Of this salt 0.1346 gram is equivalent to 1 cc. of the basic lead acetate solution, described under (a). When molasses or any other substance producing a heavy precipitate is being clarified, some dry, coarse sand should be added to break up the balls of basic lead acetate and the precipitate. (This method is to have equal weight with the use of a solution of basic lead acetate in clarifying cane, sorghum, and beet products.)

(d) *Neutral lead acetate*.—Prepare a saturated solution of neutral lead acetate and add it to the sugar solution before completing to volume. Its use is imperative when determining the reducing sugars in the solution used for polarization.

(e) *Basic lead nitrate (Herles' solution)*.—(1) Dissolve 250 grams of lead nitrate in water and make up to 500 cc. (2) Dissolve 25 grams of sodium hydroxid in water and make up to 500 cc.

Add equal amounts of (1) and (2) to the sugar solution, shake, and add more if complete precipitation has not occurred, but avoid any excess. Then complete the volume with water. When this solution is used for clarification, the factor in the Clerget determination becomes 143.5 instead of 142.66.

DETERMINATION OF SUCROSE IN THE ABSENCE OF RAFFINOSE.

(In the presence of much levulose, as in honeys and fruits products, the optical method for sucrose gives too high a result.)

14 *By Polarization Before and After Inversion with Hydrochloric Acid.—Official.*

Dissolve the normal weight (26 grams) of the substance in water, add basic lead acetate carefully, avoiding any excess, then 1-2 cc. of alumina cream, shake, and dilute to 100 metric cc., filter, rejecting the first 20 cc. of the filtrate, cover the filter with a watch glass and, when sufficient filtrate is collected, polarize in a 200 mm. tube. The reading so obtained is the direct reading (P of formula given below) or polarization before inversion. For the invert reading, remove the lead from the solution either (1) by adding anhydrous potassium oxalate, a little at a time, to the remaining solution, avoiding an excess and removing the precipitated lead by filtration; or, (2) by adding anhydrous sodium carbonate under the same conditions. Introduce 50 cc. of the lead-free filtrate into a 100 cc. flask (if sodium carbonate was used for removing the lead, neutralize carefully the excess of sodium carbonate with a few drops of dilute hydrochloric acid) and add 25 cc. of water. Then add, little by little, while rotating the flask, 5 cc. of hydrochloric acid, (sp.gr. 1.20). Heat the flask after mixing, in a water bath kept at 70°C. The temperature of the solution in the flask should reach 67°-69°C. in 2½-3 minutes. Maintain a temperature of as nearly 69°C. as possible for 7-7½ minutes, making the total time of heating 10 minutes. Remove the flask and cool the contents rapidly to 20°C. and dilute to 100 cc. Polarize this solution in a tube provided with a lateral branch and a water jacket, maintaining a temperature of 20°C. This reading must be multiplied by 2 to obtain the invert reading. If it is necessary to work at a temperature other than 20°C., which is allowable within narrow limits, the volumes must be completed and both direct and invert polarizations must be made at exactly the same temperature.

The inversion may also be accomplished as follows: (1) To 50 cc. of the clarified solution, freed from lead, add 5 cc. of hydrochloric acid (sp. gr. 1.20) and set aside for 24 hours at a temperature not below 20°C.; or, (2) If the temperature be above 25°C. set aside for 10 hours. Make up to 100 cc. at 20°C. and polarize as directed above.

Calculate sucrose by one of the following formulas:

For substances in which the invert solution contains more than 12 grams of invert sugar per 100 cc.—The following formula is to be used when substances like raw sugars are polarized:

$$S = \frac{100 (P - I)}{142.66 - \frac{T}{2}} \text{ in which}$$

S = per cent of sucrose;

P = direct reading normal solution;

I = invert reading normal solution;

T = temperature at which readings are made.

For substances in which the concentration of the invert solution is less than 12 grams per 100 cc.—The following formula, which takes into account the concentration of the sugar in solution, should be used in all other cases.

$$S = \frac{100 (P - I)}{142.66 - \frac{T}{2} - 0.0065 \left[142.66 - \frac{T}{2} - (P - I) \right]} \text{ in which}$$

S = per cent of sucrose;
 P = direct reading normal solution;
 I = invert reading normal solution;
 T = temperature.

By Polarization Before and After Inversion with Invertase.—Tentative.

15

REAGENT.

Invertase solution (Hudson Method.³)—Mix 1 kilo of pressed baker's or brewer's yeast with 1 liter of tap water and 50 cc. of toluene and keep at room temperature 2-3 days to allow autolysis to proceed to the stage of maximum inverting activity. Then add neutral lead acetate in slight excess, filter, precipitate the lead in the filtrate with hydrogen sulphid, filter again and then dialyze the filtrate thoroughly in a collodion sac. Preserve in an ice box the dialyzed solution with the addition of a little toluene to prevent the growth of micro-organisms. Note the optical activity of the invertase solution and correct the invert reading according to the amount of the solution used.

16

DETERMINATION.

Dissolve the normal weight (26 grams) of the substance in water, clarify, make up to volume, and take the direct polarization (P) as directed under 14. If lead has been used as a clarifying agent, remove the excess of lead from the filtrate, with anhydrous sodium carbonate or potassium oxalate, and filter. To 50 cc. of the filtrate in a 100 cc. flask add acetic acid, drop by drop, until the reaction is acid to litmus, add 10 cc. of the invertase solution, fill the flask with water nearly to 100 cc. and let stand in a warm place (about 40°C.) overnight. Cool and make up to 100 cc. at 20°C. Polarize at 20°C. in a 200 mm. tube. Allow the solution to remain in the tube for an hour and repeat the polarization. If there is no change from the previous reading, the inversion is complete, whereupon the reading and temperature of the solution are carefully noted. Correct the reading for the optical activity of the invertase solution and then multiply by 2. Calculate the percentage of sucrose by the following formula:

$$S = \frac{100 (P - I)}{142 - \frac{T}{2} - 0.0065 \left[142 - \frac{T}{2} - (P - I) \right]} \text{ in which}$$

S = per cent of sucrose;
 P = direct reading;
 I = invert reading;
 T = temperature at which invert reading is made.

17

DETERMINATION OF SUCROSE AND RAFFINOSE.—OFFICIAL.

(Of value chiefly in the analysis of beet products.)

If the direct reading is more than 1° higher than the per cent of sucrose as calculated by the formula given under 14, raffinose is probably present. Calculate sucrose and raffinose by the following formula of Herzfeld:

$$S = \frac{0.5124 P - I}{0.839}; \quad R = \frac{P - S}{1.852} \text{ in which}$$

P = direct reading normal solution;

I = invert reading normal solution;

S = per cent of sucrose;

R = per cent of anhydrous raffinose.

The above formula assumes that the polarizations are made at exactly 20°C. If the temperature (T) is other than 20°C., the following formula should be used:

$$S = \frac{P (0.4724 + 0.002 T) - I}{0.899 - 0.003 T}$$

Having calculated S, then $R = \frac{P - S}{1.852}$

CHEMICAL METHODS.

18 DETERMINATION OF SUCROSE FROM REDUCING SUGARS BEFORE AND AFTER INVERSION.—TENTATIVE.

Determine the reducing sugars (clarification having been effected with *neutral* lead acetate, never with basic lead acetate), as directed under 25, and calculate to invert sugar from 27. Invert the solution as directed under 14 or 16, exactly neutralize the acid, and again determine the reducing sugars, but calculate them to invert sugar from the same table as referred to above, using the invert sugar column alone. Deduct the percentage of invert sugar obtained before inversion from that obtained after inversion, and multiply the difference by 0.95, the result being the per cent of sucrose. The solutions should be diluted in both determinations so that not more than 245 mg. of invert sugar are present in the amount taken for reduction. It is important that all lead be removed from the solution with potassium oxalate before reduction.

REDUCING SUGARS.

INVERT SUGAR.

Approximate Volumetric Method for Rapid Work.—Tentative.

19

REAGENT.

Soxhlet's Modification of Fehling's Solution.—Prepare by mixing, immediately before use, equal volumes of (a) and (b).

(a) *Copper sulphate solution.*—Dissolve 34.639 grams of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water, dilute to 500 cc. and filter through prepared asbestos.

(b) *Alkaline tartrate solution.*—Dissolve 173 grams of Rochelle salts and 50 grams of sodium hydroxid in water, dilute to 500 cc., allow to stand for 2 days and filter through prepared asbestos.

20

STANDARDIZATION OF COPPER SOLUTION.

Since the factor of calculation varies with the minute details of manipulation, every operator must determine a factor for himself, using a known solution of the pure sugar that he desires to determine, and keeping the conditions the same as those used for the determination.

Standardize the solution for invert sugar in the following manner:

Dissolve 4.75 grams of pure sucrose in 75 cc. of water, add 5 cc. of hydrochloric acid (sp. gr. 1.20) and invert as directed under 14. Neutralize the acid with sodium hydroxid solution and dilute to 1 liter. Ten cc. of this solution contain 0.050 gram of invert sugar, which should reduce 10 cc. of the reagent. The strength of the copper solution should never be taken as a constant, but should be checked against the sugar.

21

DETERMINATION.

Place 10 cc. of the reagent in a large test tube and add 10 cc. of water. Heat to boiling, and add gradually small portions of the solution of the material to be tested until the copper has been completely reduced, boiling after each addition to complete the reaction. Two minutes' boiling is required for complete reduction when the full amount of sugar solution has been added in one portion. When the end is nearly reached and the amount of sugar solution to be added can no longer be judged by the color of the solution, remove a small portion of the liquid and filter rapidly into a small porcelain crucible or on a test plate; acidify with dilute acetic acid, and test for copper with dilute potassium ferrocyanid solution. The sugar solution should be of such strength as will give a burette reading of 15-20 cc., and the number of successive additions should be as small as possible.

Soxhlet Volumetric Method.—Tentative.

22

REAGENT.

The reagent used is described under 19.

23

DETERMINATION.

Make a preliminary titration to determine the approximate percentage of reducing sugar in the material under examination. Prepare a solution which contains approximately 1% of reducing sugar. Place in a beaker 100 cc. of the reagent and approximately the amount of the sugar solution for its complete reduction. Boil for 2 minutes. Filter through a folded filter and test a portion of the filtrate for copper by use of dilute acetic acid and dilute potassium ferrocyanid solution. Repeat, varying the volume of sugar solution, until 2 successive amounts are found which differ by 0.1 cc., one giving complete reduction and the other leaving a small amount of copper in solution. The mean of these 2 readings is taken as the volume of the solution required for the complete precipitation of 100 cc. of the reagent.

Under these conditions 100 cc. of the reagent require 0.494 gram of invert sugar for complete reduction. Calculate the percentage by the following formula:

V = the volume of the sugar solution required for the complete reduction of 100 cc. of the reagent;

W = the weight of the sample in 1 cc. of the sugar solution;

$$\frac{100 \times 0.494}{VW} = \text{per cent of invert sugar.}$$

GRAVIMETRIC METHODS.

Munson and Walker General Method.—Tentative.

24

REAGENTS.

(a) *Asbestos*.—Digest the asbestos, which should be the amphibole variety, with dilute hydrochloric acid (1 to 3) for 2-3 days. Wash free from acid, digest for a

similar period with 10% sodium hydroxid solution, and then treat for a few hours with hot alkaline tartrate solution (old alkaline tartrate solutions that have stood for some time may be used for this purpose) of the strength employed in sugar determinations. Then wash the asbestos free from alkali, digest for several hours with dilute nitric acid (1 to 3) and, after washing free from acid, shake with water into a fine pulp. In preparing the Gooch crucible, make a film of asbestos $\frac{1}{2}$ inch thick and wash thoroughly with water to remove fine particles of asbestos. If the precipitated cuprous oxid is to be weighed as such, wash the crucible with 10 cc. of alcohol, then with 10 cc. of ether, dry for 30 minutes at 100°C., cool in a desiccator and weigh.

(b) The solution used is described under 19.

25**PRECIPITATION OF CUPROUS OXID.**

Transfer 25 cc. each of the copper sulphate and alkaline tartrate solutions to a 400 cc. beaker of alkali-resisting glass and add 50 cc. of reducing sugar solution, or, if a smaller volume of sugar solution is used, add water to make the final volume 100 cc. Heat the beaker upon an asbestos gauze over a Bunsen burner, regulate the flame so that boiling begins in 4 minutes, and continue the boiling for exactly 2 minutes. (It is important that these directions be strictly observed and, in order to regulate the burner for this purpose, it is advisable to make preliminary tests, using 50 cc. of the reagent and 50 cc. of water before proceeding with the actual determination.) Keep the beaker covered with a watch glass during the heating. Filter the cuprous oxid at once on an asbestos mat in a porcelain Gooch crucible, using suction. Wash the cuprous oxid thoroughly with water at a temperature of about 60°C., and either weigh directly as cuprous oxid as in 26, or, determine the amount of reduced copper by one of the methods under 29-34, respectively. Conduct a blank determination, using 50 cc. of the reagent and 50 cc. of water, and, if the weight of cuprous oxid obtained exceeds 0.5 mg., correct the result of the reducing sugar determination accordingly. The alkaline tartrate solution deteriorates on standing and the amount of cuprous oxid obtained in the blank increases.

DETERMINATION OF REDUCED COPPER.**26****I. Direct Weighing of Cuprous Oxid.—Tentative.**

Prepare a Gooch as directed under 24 (a).

Collect the precipitated cuprous oxid on the mat, as directed under 25, wash thoroughly with hot water, then with 10 cc. of alcohol, and finally with 10 cc. of ether. Dry the precipitate for 30 minutes in a water oven at the temperature of boiling water; cool and weigh. Calculate the weight of metallic copper. Obtain from 27 the weight of invert sugar equivalent to the weight of copper found.

This method should be used only for determinations in pure sugar solutions. In all other products the copper of the cuprous oxid should be determined by one of the following methods, since the cuprous oxid is very apt to be contaminated with foreign matter.

The number of milligrams of copper reduced by a given amount of reducing sugar differs when sucrose is present and when it is absent. In the tables the absence of sucrose is assumed except in the two columns under invert sugar, where one for mixtures of invert sugar and sucrose containing 0.4 gram of total sugar in 50 cc. of solution, and one for invert sugar and sucrose when the 50 cc. of solution contains 2 grams of total sugar are given, in addition to the column for invert sugar alone.

27

TABLE 1.—MUNSON AND WALKER'S TABLE.

For calculating dextrose, invert sugar alone, invert sugar in the presence of sucrose (0.4 gram and 2 grams total sugar), lactose (two forms), and maltose (anhydrous and crystallized).

[Expressed in milligrams.]

CUPREOUS OXID (Cu ₂ O)	COPPER (Cu)	DEXTRIN (d-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPREOUS OXID (Cu ₂ O)
				0.4 gram total sugar	2 grams total sugar	C ₆ H ₁₂ O ₆	C ₆ H ₁₂ O ₆ ·H ₂ O	C ₆ H ₁₂ O ₆	C ₆ H ₁₂ O ₆ ·H ₂ O	
10	8.9	4.0	4.5	1.6	3.8	4.0	5.9	6.2	10
11	9.8	4.5	5.0	2.1	4.5	4.7	6.7	7.0	11
12	10.7	4.9	5.4	2.5	5.1	5.4	7.5	7.9	12
13	11.5	5.3	5.8	3.0	5.8	6.1	8.3	8.7	13
14	12.4	5.7	6.3	3.4	6.4	6.8	9.1	9.5	14
15	13.3	6.2	6.7	3.9	7.1	7.5	9.9	10.4	15
16	14.2	6.6	7.2	4.3	7.8	8.2	10.6	11.2	16
17	15.1	7.0	7.6	4.8	8.4	8.9	11.4	12.0	17
18	16.0	7.5	8.1	5.2	9.1	9.5	12.2	12.9	18
19	16.9	7.9	8.5	5.7	9.7	10.2	13.0	13.7	19
20	17.8	8.3	8.9	6.1	10.4	10.9	13.8	14.6	20
21	18.7	8.7	9.4	6.6	11.0	11.6	14.6	15.4	21
22	19.5	9.2	9.8	7.0	11.7	12.3	15.4	16.2	22
23	20.4	9.6	10.3	7.5	12.3	13.0	16.2	17.1	23
24	21.3	10.0	10.7	7.9	13.0	13.7	17.0	17.9	24
25	22.2	10.5	11.2	8.4	13.7	14.4	17.8	18.7	25
26	23.1	10.9	11.6	8.8	14.3	15.1	18.6	19.6	26
27	24.0	11.3	12.0	9.3	15.0	15.8	19.4	20.4	27
28	24.9	11.8	12.5	9.7	15.6	16.5	20.2	21.2	28
29	25.8	12.2	12.9	10.2	16.3	17.1	21.0	22.1	29
30	26.6	12.6	13.4	10.7	4.3	16.9	17.8	21.8	22.9	30
31	27.5	13.1	13.8	11.1	4.7	17.6	18.5	22.6	23.7	31
32	28.4	13.5	14.3	11.6	5.2	18.3	19.2	23.3	24.6	32
33	29.3	13.9	14.7	12.0	5.6	18.9	19.9	24.1	25.4	33
34	30.2	14.3	15.2	12.5	6.1	19.6	20.6	24.9	26.2	34
35	31.1	14.8	15.6	12.9	6.5	20.2	21.3	25.7	27.1	35
36	32.0	15.2	16.1	13.4	7.0	20.9	22.0	26.5	27.9	36
37	32.9	15.6	16.5	13.8	7.4	21.5	22.7	27.3	28.7	37
38	33.8	16.1	16.9	14.3	7.9	22.2	23.4	28.1	29.6	38
39	34.6	16.5	17.4	14.7	8.4	22.8	24.1	28.9	30.4	39
40	35.5	16.9	17.8	15.2	8.8	23.5	24.8	29.7	31.3	40
41	36.4	17.4	18.3	15.6	9.3	24.2	25.4	30.5	32.1	41
42	37.3	17.8	18.7	16.1	9.7	24.8	26.1	31.3	32.9	42
43	38.2	18.2	19.2	16.6	10.2	25.5	26.8	32.1	33.8	43
44	39.1	18.7	19.6	17.0	10.7	26.1	27.5	32.9	34.6	44
45	40.0	19.1	20.1	17.5	11.1	26.8	28.2	33.7	35.4	45
46	40.9	19.6	20.5	17.9	11.6	27.4	28.9	34.4	36.3	46
47	41.7	20.0	21.0	18.4	12.0	28.1	29.6	35.2	37.1	47
48	42.6	20.4	21.4	18.8	12.5	28.7	30.3	36.0	37.9	48
49	43.5	20.9	21.9	19.3	12.9	29.4	31.0	36.8	38.8	49
50	44.4	21.3	22.3	19.7	13.4	30.1	31.7	37.6	39.6	50
51	45.3	21.7	22.8	20.2	13.9	30.7	32.4	38.4	40.4	51
52	46.2	22.2	23.2	20.7	14.3	31.4	33.0	39.2	41.3	52
53	47.1	22.6	23.7	21.1	14.8	32.1	33.7	40.0	42.1	53
54	48.0	23.0	24.1	21.6	15.2	32.7	34.4	40.8	42.9	54
55	48.9	23.5	24.6	22.0	15.7	33.4	35.1	41.6	43.8	55
56	49.7	23.9	25.0	22.5	16.2	34.0	35.8	42.4	44.6	56
57	50.6	24.3	25.5	22.9	16.6	34.7	36.5	43.2	45.4	57
58	51.5	24.8	25.9	23.4	17.1	35.4	37.2	44.0	46.3	58
59	52.4	25.2	26.4	23.9	17.5	36.0	37.9	44.8	47.1	59
60	53.3	25.6	26.8	24.3	18.0	36.7	38.6	45.6	48.0	60
61	54.2	26.1	27.3	24.8	18.5	37.3	39.3	46.3	48.8	61
62	55.1	26.5	27.7	25.2	18.9	38.0	40.0	47.1	49.6	62
63	56.0	27.0	28.2	25.7	19.4	38.6	40.7	47.9	50.5	63
64	56.8	27.4	28.6	26.2	19.8	39.3	41.4	48.7	51.3	64

27

TABLE 1.—MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu_2O)	COPPER (Cu)	DEXTRINE (d-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu_2O)
				0.4 gram total sugar	2 grams total sugar	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$	$\text{C}_{12}\text{H}_{22}\text{O}_{11}\cdot\text{H}_2\text{O}$	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$	$\text{C}_{12}\text{H}_{22}\text{O}_{11}\cdot\text{H}_2\text{O}$	
65	57.7	27.8	29.1	26.6	20.3	40.0	42.1	49.5	52.1	65
66	58.6	28.3	29.5	27.1	20.8	40.6	42.8	50.3	53.0	66
67	59.5	28.7	30.0	27.5	21.2	41.3	43.5	51.1	53.8	67
68	60.4	29.2	30.4	28.0	21.7	41.9	44.2	51.9	54.6	68
69	61.3	29.6	30.9	28.5	22.2	42.6	44.8	52.7	55.5	69
70	62.2	30.0	31.3	28.9	22.6	43.3	45.5	53.5	56.3	70
71	63.1	30.5	31.8	29.4	23.1	43.9	46.2	54.3	57.1	71
72	64.0	30.9	32.3	29.8	23.5	44.6	46.9	55.1	58.0	72
73	64.8	31.4	32.7	30.3	24.0	45.2	47.6	55.9	58.8	73
74	65.7	31.8	33.2	30.8	24.5	45.9	48.3	56.7	59.6	74
75	66.6	32.2	33.6	31.2	24.9	46.6	49.0	57.5	60.5	75
76	67.5	32.7	34.1	31.7	25.4	47.2	49.7	58.2	61.3	76
77	68.4	33.1	34.5	32.1	25.9	47.9	50.4	59.0	62.1	77
78	69.3	33.6	35.0	32.6	26.3	48.5	51.1	59.8	63.0	78
79	70.2	34.0	35.4	33.1	26.8	49.2	51.8	60.6	63.8	79
80	71.1	34.4	35.9	33.5	27.3	49.9	52.5	61.4	64.6	80
81	71.9	34.9	36.3	34.0	27.7	50.5	53.2	62.2	65.5	81
82	72.8	35.3	36.8	34.5	28.2	51.2	53.9	63.0	66.3	82
83	73.7	35.8	37.3	34.9	28.6	51.8	54.6	63.8	67.1	83
84	74.6	36.2	37.7	35.4	29.1	52.5	55.3	64.6	68.0	84
85	75.5	36.7	38.2	35.8	29.6	53.1	56.0	65.4	68.8	85
86	76.4	37.1	38.6	36.3	30.0	53.8	56.6	66.2	69.7	86
87	77.3	37.5	39.1	36.8	30.5	54.5	57.3	67.0	70.5	87
88	78.2	38.0	39.5	37.2	31.0	55.1	58.0	67.8	71.3	88
89	79.1	38.4	40.0	37.7	31.4	55.8	58.7	68.5	72.2	89
90	79.9	38.9	40.4	38.2	31.9	56.4	59.4	69.3	73.0	90
91	80.8	39.3	40.9	38.6	32.4	57.1	60.1	70.1	73.8	91
92	81.7	39.8	41.4	39.1	32.8	57.8	60.8	70.9	74.7	92
93	82.6	40.2	41.8	39.6	33.3	58.4	61.5	71.7	75.5	93
94	83.5	40.6	42.3	40.0	33.8	59.1	62.2	72.5	76.3	94
95	84.4	41.1	42.7	40.5	34.2	59.7	62.9	73.3	77.2	95
96	85.3	41.5	43.2	41.0	34.7	60.4	63.6	74.1	78.0	96
97	86.2	42.0	43.7	41.4	35.2	61.1	64.3	74.9	78.8	97
98	87.1	42.4	44.1	41.9	35.6	61.7	65.0	75.7	79.7	98
99	87.9	42.9	44.6	42.4	36.1	62.4	65.7	76.5	80.5	99
100	88.8	43.3	45.0	42.8	36.6	63.0	66.4	77.3	81.3	100
101	89.7	43.8	45.5	43.3	37.0	63.7	67.1	78.1	82.2	101
102	90.6	44.2	46.0	43.8	37.5	64.4	67.8	78.8	83.0	102
103	91.5	44.7	46.4	44.2	38.0	65.0	68.5	79.6	83.8	103
104	92.4	45.1	46.9	44.7	38.5	65.7	69.1	80.4	84.7	104
105	93.3	45.5	47.3	45.2	38.9	66.4	69.8	81.2	85.5	105
106	94.2	46.0	47.8	45.6	39.4	67.0	70.5	82.0	86.3	106
107	95.0	46.4	48.3	46.1	39.9	67.7	71.2	82.8	87.2	107
108	95.9	46.9	48.7	46.6	40.3	68.3	71.9	83.6	88.0	108
109	96.8	47.3	49.2	47.0	40.8	69.0	72.6	84.4	88.8	109
110	97.7	47.8	49.6	47.5	41.3	69.7	73.3	85.2	89.7	110
111	98.6	48.2	50.1	48.0	41.7	70.3	74.0	86.0	90.5	111
112	99.5	48.7	50.6	48.4	42.2	71.0	74.7	86.8	91.3	112
113	100.4	49.1	51.0	48.9	42.7	71.6	75.4	87.6	92.2	113
114	101.3	49.6	51.5	49.4	43.2	72.3	76.1	88.4	93.0	114
115	102.2	50.0	51.9	49.8	43.6	73.0	76.8	89.2	93.9	115
116	103.0	50.5	52.4	50.3	44.1	73.6	77.5	90.0	94.7	116
117	103.9	50.9	52.9	50.8	44.6	74.3	78.2	90.7	95.5	117
118	104.8	51.4	53.3	51.2	45.0	75.0	78.9	91.5	96.4	118
119	105.7	51.8	53.8	51.7	45.5	75.6	79.6	92.3	97.2	119

TABLE 1.—MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CURIOUS OXID (Cu ₂ O)	COPPER (Cu)	DEXTROSE (d-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CURIOUS OXID (Cu ₂ O)
				0.4 gram total sugar	2 grams total sugar	CuH ₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	
120	106.6	53.8	54.3	52.2	46.0	76.3	80.3	93.1	98.0	130
121	107.5	53.7	54.7	52.7	46.5	76.9	81.0	93.9	98.9	131
122	108.4	53.2	55.2	53.1	46.9	77.6	81.7	94.7	99.7	132
123	100.3	53.6	55.7	53.6	47.4	78.3	82.4	95.5	100.5	133
124	110.1	54.1	56.1	54.1	47.9	78.9	83.1	96.3	101.4	134
125	111.0	54.5	56.6	54.5	48.3	79.6	83.8	97.1	102.2	135
126	111.9	55.0	57.0	55.0	48.8	80.3	84.5	97.9	103.0	136
127	112.8	55.4	57.5	55.5	49.3	80.9	85.2	98.7	103.9	137
128	113.7	55.9	58.0	55.9	49.8	81.6	85.9	99.4	104.7	138
129	114.6	56.3	58.4	56.4	50.2	82.2	86.6	100.2	105.5	139
130	115.5	56.8	58.9	56.9	50.7	82.9	87.3	101.0	106.4	140
131	116.4	57.2	59.4	57.4	51.2	83.6	88.0	101.8	107.2	141
132	117.3	57.7	59.8	57.8	51.7	84.2	88.7	102.6	108.0	142
133	118.1	58.1	60.3	58.3	52.1	84.9	89.4	103.4	108.9	143
134	119.0	58.6	60.8	58.8	52.6	85.5	90.1	104.2	109.7	144
135	119.9	59.0	61.2	59.3	53.1	86.2	90.8	105.0	110.5	145
136	120.8	59.5	61.7	59.7	53.6	86.9	91.5	105.8	111.4	146
137	121.7	60.0	62.2	60.2	54.0	87.5	92.1	106.6	112.2	147
138	122.6	60.4	62.6	60.7	54.5	88.2	92.8	107.4	113.0	148
139	123.5	60.9	63.1	61.2	55.0	88.9	93.5	108.2	113.9	149
140	124.4	61.3	63.6	61.6	55.5	89.5	94.2	109.0	114.7	150
141	125.2	61.8	64.0	62.1	55.9	90.2	94.9	109.8	115.5	151
142	126.1	62.3	64.5	62.6	56.4	90.8	95.6	110.5	116.4	152
143	127.0	62.7	65.0	63.1	56.9	91.5	96.3	111.3	117.2	153
144	127.9	63.1	65.4	63.5	57.4	92.2	97.0	112.1	118.0	154
145	128.8	63.6	65.9	64.0	57.8	92.8	97.7	112.9	118.9	155
146	129.7	64.0	66.4	64.5	58.3	93.5	98.4	113.7	119.7	156
147	130.6	64.5	66.9	65.0	58.8	94.2	99.1	114.5	120.5	157
148	131.5	65.0	67.3	65.4	59.3	94.8	99.8	115.3	121.4	158
149	132.4	65.4	67.8	65.9	59.7	95.5	100.5	116.1	122.2	159
150	133.3	65.9	68.3	66.4	60.2	96.1	101.2	116.9	123.0	160
151	134.1	66.3	68.7	66.9	60.7	96.8	101.9	117.7	123.9	161
152	135.0	66.8	69.2	67.3	61.2	97.5	102.6	118.5	124.7	162
153	135.9	67.2	69.7	67.8	61.7	98.1	103.3	119.3	125.5	163
154	136.8	67.7	70.1	68.3	62.1	98.8	104.0	120.0	126.4	164
155	137.7	68.2	70.6	68.8	62.6	99.5	104.7	120.8	127.2	165
156	138.6	68.6	71.1	69.2	63.1	100.1	105.4	121.6	128.0	166
157	139.5	69.1	71.6	69.7	63.6	100.8	106.1	122.4	128.9	167
158	140.3	69.5	72.0	70.2	64.1	101.5	106.8	123.2	129.7	168
159	141.2	70.0	72.5	70.7	64.5	102.1	107.5	124.0	130.5	169
160	142.1	70.4	73.0	71.2	65.0	102.8	108.2	124.8	131.4	170
161	143.0	70.9	73.4	71.6	65.5	103.4	108.9	125.6	132.2	171
162	143.9	71.4	73.9	72.1	66.0	104.1	109.6	126.4	133.0	172
163	144.8	71.8	74.4	72.6	66.5	104.8	110.3	127.2	133.9	173
164	145.7	72.3	74.9	73.1	66.9	105.4	111.0	128.0	134.7	174
165	146.6	72.8	75.3	73.6	67.4	106.1	111.7	128.8	135.5	175
166	147.5	73.2	75.8	74.0	67.9	106.8	112.4	129.6	136.4	176
167	148.3	73.7	76.3	74.5	68.4	107.4	113.1	130.3	137.2	177
168	149.2	74.1	76.8	75.0	68.9	108.1	113.8	131.1	138.0	178
169	150.1	74.6	77.2	75.5	69.3	108.8	114.5	131.9	138.9	179
170	151.0	75.1	77.7	76.0	69.8	109.4	115.2	132.7	139.7	180
171	151.9	75.5	78.2	76.4	70.3	110.1	115.9	133.5	140.5	181
172	152.8	76.0	78.7	76.9	70.8	110.8	116.6	134.3	141.4	182
173	153.7	76.4	79.1	77.4	71.3	111.4	117.3	135.1	142.2	183
174	154.6	76.9	79.6	77.9	71.7	112.1	118.0	135.9	143.0	184

TABLE 1.—MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPREOUS OXID (Cu ₂ O)	COPPER (Cu)	DEXTROSE (d-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPREOUS OXID (Cu ₂ O)
				0.4 gram sugar total	2 grams sugar total	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	
175	155.5	77.4	80.1	78.4	72.2	112.8	118.7	136.7	143.9	175
176	156.3	77.8	80.6	78.8	72.7	113.4	119.4	137.5	144.7	176
177	157.2	78.3	81.0	79.3	73.2	114.1	120.1	138.3	145.5	177
178	158.1	78.8	81.5	79.8	73.7	114.8	120.8	139.1	146.4	178
179	159.0	79.2	82.0	80.3	74.2	115.4	121.5	139.8	147.2	179
180	159.9	79.7	82.5	80.8	74.6	116.1	122.2	140.6	148.0	180
181	160.8	80.1	82.9	81.3	75.1	116.7	122.9	141.4	148.9	181
182	161.7	80.6	83.4	81.7	75.6	117.4	123.6	142.2	149.7	182
183	162.6	81.1	83.9	82.2	76.1	118.1	124.3	143.0	150.5	183
184	163.4	81.5	84.4	82.7	76.6	118.7	125.0	143.8	151.4	184
185	164.3	82.0	84.9	83.2	77.1	119.4	125.7	144.6	152.2	185
186	165.2	82.5	85.3	83.7	77.6	120.1	126.4	145.4	153.0	186
187	166.1	82.9	85.8	84.2	78.0	120.7	127.1	146.2	153.9	187
188	167.0	83.4	86.3	84.6	78.5	121.4	127.8	147.0	154.7	188
189	167.9	83.9	86.8	85.1	79.0	122.1	128.5	147.8	155.5	189
190	168.8	84.3	87.2	85.6	79.5	122.7	129.2	148.6	156.4	190
191	169.7	84.8	87.7	86.1	80.0	123.4	129.9	149.3	157.2	191
192	170.5	85.3	88.2	86.6	80.5	124.1	130.6	150.1	158.0	192
193	171.4	85.7	88.7	87.1	81.0	124.7	131.3	150.9	158.9	193
194	172.3	86.2	89.2	87.6	81.4	125.4	132.0	151.7	159.7	194
195	173.2	86.7	89.6	88.0	81.9	126.1	132.7	152.5	160.5	195
196	174.1	87.1	90.1	88.5	82.4	126.7	133.4	153.3	161.4	196
197	175.0	87.6	90.6	89.0	82.9	127.4	134.1	154.1	162.2	197
198	175.9	88.1	91.1	89.5	83.4	128.1	134.8	154.9	163.0	198
199	176.8	88.5	91.6	90.0	83.9	128.7	135.5	155.7	163.9	199
200	177.7	89.0	92.0	90.5	84.4	129.4	136.2	156.5	164.7	200
201	178.5	89.5	92.5	91.0	84.8	130.0	136.9	157.3	165.5	201
202	179.4	89.9	93.0	91.4	85.3	130.7	137.6	158.1	166.4	202
203	180.3	90.4	93.5	91.9	85.8	131.4	138.3	158.8	167.2	203
204	181.2	90.9	94.0	92.4	86.3	132.0	139.0	159.6	168.0	204
205	182.1	91.4	94.5	92.9	86.8	132.7	139.7	160.4	168.9	205
206	183.0	91.8	94.9	93.4	87.3	133.4	140.4	161.2	169.7	206
207	183.9	92.3	95.4	93.9	87.8	134.0	141.1	162.0	170.5	207
208	184.8	92.8	95.9	94.4	88.3	134.7	141.8	162.8	171.4	208
209	185.6	93.2	96.4	94.9	88.8	135.4	142.5	163.6	172.2	209
210	186.5	93.7	96.9	95.4	89.2	136.0	143.2	164.4	173.0	210
211	187.4	94.2	97.4	95.8	89.7	136.7	143.9	165.2	173.8	211
212	188.3	94.6	97.8	96.3	90.2	137.4	144.6	166.0	174.7	212
213	189.2	95.1	98.3	96.8	90.7	138.0	145.3	166.8	175.5	213
214	190.1	95.6	98.8	97.3	91.2	138.7	146.0	167.5	176.4	214
215	191.0	96.1	99.3	97.8	91.7	139.4	146.7	168.3	177.2	215
216	191.9	96.5	99.8	98.3	92.2	140.0	147.4	169.1	178.0	216
217	192.8	97.0	100.3	98.8	92.7	140.7	148.1	169.9	178.9	217
218	193.6	97.5	100.8	99.3	93.2	141.4	148.8	170.7	179.7	218
219	194.5	98.0	101.2	99.8	93.7	142.0	149.5	171.5	180.5	219
220	195.4	98.4	101.7	100.3	94.2	142.7	150.2	172.3	181.4	220
221	196.3	98.9	102.2	100.8	94.7	143.4	150.9	173.1	182.2	221
222	197.2	99.4	102.7	101.2	95.1	144.0	151.6	173.9	183.0	222
223	198.1	99.9	103.2	101.7	95.6	144.7	152.3	174.7	183.9	223
224	199.0	100.3	103.7	102.2	96.1	145.4	153.0	175.5	184.7	224
225	199.9	100.8	104.2	102.7	96.6	146.0	153.7	176.2	185.5	225
226	200.7	101.3	104.6	103.2	97.1	146.7	154.4	177.0	186.4	226
227	201.6	101.8	105.1	103.7	97.6	147.4	155.1	177.8	187.2	227
228	202.5	102.2	105.6	104.2	98.1	148.0	155.8	178.6	188.0	228
229	203.4	102.7	106.1	104.7	98.6	148.7	156.5	179.4	188.8	229

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TABLE 1.—MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu ₂ O)	COPPER (Cu)	DEXTROSE (d-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu ₂ O)
				0.4 gram sugar	2 grams sugar	CuH ₁₂ O ₁₁	CuH ₁₂ O ₁₁ H ₂ O	CuH ₁₂ O ₁₁	CuH ₁₂ O ₁₁ H ₂ O	
230	204.3	103.2	106.6	105.2	99.1	149.4	157.2	180.2	189.7	230
231	205.2	103.7	107.1	105.7	99.6	150.0	157.9	181.0	190.5	231
232	206.1	104.1	107.6	106.2	100.1	150.7	158.6	181.8	191.3	232
233	207.0	104.6	108.1	106.7	100.6	151.4	159.3	182.6	192.2	233
234	207.9	105.1	108.6	107.2	101.1	152.0	160.0	183.4	193.0	234
235	208.7	105.6	109.1	107.7	101.6	152.7	160.7	184.2	193.8	235
236	209.6	106.0	109.5	108.2	102.1	153.4	161.4	184.9	194.7	236
237	210.5	106.5	110.0	108.7	102.6	154.0	162.1	185.7	195.5	237
238	211.4	107.0	110.5	109.2	103.1	154.7	162.8	186.5	196.3	238
239	212.3	107.5	111.0	109.6	103.6	155.4	163.5	187.3	197.2	239
240	213.2	108.0	111.5	110.1	104.0	156.1	164.3	188.1	198.0	240
241	214.1	108.4	112.0	110.6	104.5	156.7	165.0	188.9	198.8	241
242	215.0	108.9	112.5	111.1	105.0	157.4	165.7	189.7	199.7	242
243	215.8	109.4	113.0	111.6	105.5	158.1	166.4	190.5	200.5	243
244	216.7	109.9	113.5	112.1	106.0	158.7	167.1	191.3	201.3	244
245	217.6	110.4	114.0	112.6	106.5	159.4	167.8	192.1	202.2	245
246	218.5	110.8	114.5	113.1	107.0	160.1	168.5	192.9	203.0	246
247	219.4	111.3	115.0	113.6	107.5	160.7	169.2	193.6	203.8	247
248	220.3	111.8	115.4	114.1	108.0	161.4	169.9	194.4	204.7	248
249	221.2	112.3	115.9	114.6	108.5	162.1	170.6	195.2	205.5	249
250	222.1	112.8	116.4	115.1	109.0	162.7	171.3	196.0	206.3	250
251	223.0	113.2	116.9	115.6	109.5	163.4	172.0	196.8	207.2	251
252	223.8	113.7	117.4	116.1	110.0	164.1	172.7	197.6	208.0	252
253	224.7	114.2	117.9	116.6	110.5	164.7	173.4	198.4	208.8	253
254	225.6	114.7	118.4	117.1	111.0	165.4	174.1	199.2	209.7	254
255	226.5	115.2	118.9	117.6	111.5	166.1	174.8	200.0	210.5	255
256	227.4	115.7	119.4	118.1	112.0	166.8	175.5	200.8	211.3	256
257	228.3	116.1	119.9	118.6	112.5	167.4	176.2	201.6	212.2	257
258	229.2	116.6	120.4	119.1	113.0	168.1	176.9	202.3	213.0	258
259	230.1	117.1	120.9	119.6	113.5	168.8	177.6	203.1	213.8	259
260	231.0	117.6	121.4	120.1	114.0	169.4	178.3	203.9	214.7	260
261	231.8	118.1	121.9	120.6	114.5	170.1	179.0	204.7	215.5	261
262	232.7	118.6	122.4	121.1	115.0	170.8	179.8	205.5	216.3	262
263	233.6	119.0	122.9	121.6	115.5	171.4	180.5	206.3	217.2	263
264	234.5	119.5	123.4	122.1	116.0	172.1	181.2	207.1	218.0	264
265	235.4	120.0	123.9	122.6	116.5	172.8	181.9	207.9	218.8	265
266	236.3	120.5	124.4	123.1	117.0	173.5	182.6	208.7	219.7	266
267	237.2	121.0	124.9	123.6	117.5	174.1	183.3	209.5	220.5	267
268	238.1	121.5	125.4	124.1	118.0	174.8	184.0	210.3	221.3	268
269	238.9	122.0	125.9	124.6	118.5	175.5	184.7	211.0	222.1	269
270	239.8	122.5	126.4	125.1	119.0	176.1	185.4	211.8	223.0	270
271	240.7	122.9	126.9	125.6	119.5	176.8	186.1	212.6	223.8	271
272	241.6	123.4	127.4	126.2	120.0	177.5	186.8	213.4	224.6	272
273	242.5	123.9	127.9	126.7	120.5	178.1	187.5	214.2	225.5	273
274	243.4	124.4	128.4	127.2	121.1	178.8	188.2	215.0	226.3	274
275	244.3	124.9	128.9	127.7	121.6	179.5	188.9	215.8	227.1	275
276	245.2	125.4	129.4	128.2	122.1	180.2	189.6	216.6	228.0	276
277	246.1	125.9	129.9	128.7	122.6	180.8	190.3	217.4	228.8	277
278	246.9	126.4	130.4	129.2	123.1	181.5	191.0	218.2	229.6	278
279	247.8	126.9	130.9	129.7	123.6	182.2	191.7	218.9	230.5	279
280	248.7	127.3	131.4	130.2	124.1	182.8	192.4	219.7	231.3	280
281	249.6	127.8	131.9	130.7	124.6	183.5	193.1	220.5	232.1	281
282	250.5	128.3	132.4	131.2	125.1	184.2	193.9	221.3	233.0	282
283	251.4	128.8	132.9	131.7	125.6	184.8	194.6	222.1	233.8	283
284	252.3	129.3	133.4	132.2	126.1	185.5	195.3	222.9	234.6	284

TABLE 1.—MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXIDE (Cu ₂ O)	COPPER (Cu)	DEXTROSE (β-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXIDE (Cu ₂ O)
				0.4 gram sugar	2 grams sugar	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	
285	252.2	129.8	133.9	132.7	126.6	186.2	196.0	223.7	235.5	285
286	254.0	130.3	134.4	133.2	127.1	186.9	196.7	224.5	236.3	286
287	254.9	130.8	134.9	133.7	127.6	187.5	197.4	225.3	237.1	287
288	255.8	131.3	135.4	134.3	128.1	188.2	198.1	226.1	238.0	288
289	256.7	131.8	135.9	134.8	128.6	188.9	198.8	226.9	238.8	289
290	257.6	132.3	136.4	135.3	129.2	189.5	199.5	227.6	239.6	290
291	258.5	132.7	136.9	135.8	129.7	190.2	200.2	228.4	240.5	291
292	259.4	133.2	137.4	136.3	130.2	190.9	200.9	229.2	241.3	292
293	260.3	133.7	137.9	136.8	130.7	191.5	201.6	230.0	242.1	293
294	261.2	134.2	138.4	137.3	131.2	192.2	202.3	230.8	242.9	294
295	262.0	134.7	138.9	137.8	131.7	192.9	203.0	231.6	243.8	295
296	262.9	135.2	139.4	138.3	132.2	193.6	203.7	232.4	244.6	296
297	263.8	135.7	140.0	138.8	132.7	194.2	204.4	233.2	245.4	297
298	264.7	136.2	140.5	139.4	133.2	194.9	205.1	234.0	246.3	298
299	265.6	136.7	141.0	139.9	133.7	195.6	205.8	234.8	247.1	299
300	266.5	137.2	141.5	140.4	134.2	196.2	206.6	235.5	247.9	300
301	267.4	137.7	142.0	140.9	134.8	196.9	207.3	236.3	248.8	301
302	268.3	138.2	142.5	141.4	135.3	197.6	208.0	237.1	249.6	302
303	269.1	138.7	143.0	141.9	135.8	198.3	208.7	237.9	250.4	303
304	270.0	139.2	143.5	142.4	136.3	198.9	209.4	238.7	251.3	304
305	270.9	139.7	144.0	142.9	136.8	199.6	210.1	239.5	252.1	305
306	271.8	140.2	144.5	143.4	137.3	200.3	210.8	240.3	252.9	306
307	272.7	140.7	145.0	144.0	137.8	201.0	211.5	241.1	253.8	307
308	273.6	141.2	145.5	144.5	138.3	201.6	212.2	241.9	254.6	308
309	274.5	141.7	146.1	145.0	138.8	202.3	212.9	242.7	255.4	309
310	275.4	142.2	146.6	145.5	139.4	203.0	213.7	243.5	256.3	310
311	276.3	142.7	147.1	146.0	139.9	203.6	214.4	244.2	257.1	311
312	277.1	143.2	147.6	146.5	140.4	204.3	215.1	245.0	257.9	312
313	278.0	143.7	148.1	147.0	140.9	205.0	215.8	245.8	258.8	313
314	278.9	144.2	148.6	147.6	141.4	205.7	216.5	246.6	259.6	314
315	279.8	144.7	149.1	148.1	141.9	206.3	217.2	247.4	260.4	315
316	280.7	145.2	149.6	148.6	142.4	207.0	217.9	248.2	261.2	316
317	281.6	145.7	150.1	149.1	143.0	207.7	218.6	249.0	262.1	317
318	282.5	146.2	150.7	149.6	143.5	208.4	219.3	249.8	262.9	318
319	283.4	146.7	151.2	150.1	144.0	209.0	220.0	250.6	263.7	319
320	284.2	147.2	151.7	150.7	144.5	209.7	220.7	251.3	264.6	320
321	285.1	147.7	152.2	151.2	145.0	210.4	221.4	252.1	265.4	321
322	286.0	148.2	152.7	151.7	145.5	211.0	222.2	252.9	266.2	322
323	286.9	148.7	153.2	152.2	146.0	211.7	222.9	253.7	267.1	323
324	287.8	149.2	153.7	152.7	146.6	212.4	223.6	254.5	267.9	324
325	288.7	149.7	154.3	153.2	147.1	213.1	224.3	255.3	268.7	325
326	289.6	150.2	154.8	153.8	147.6	213.7	225.0	256.1	269.6	326
327	290.5	150.7	155.3	154.3	148.1	214.4	225.7	256.9	270.4	327
328	291.4	151.2	155.8	154.8	148.6	215.1	226.4	257.7	271.2	328
329	292.2	151.7	156.3	155.3	149.1	215.8	227.1	258.5	272.1	329
330	293.1	152.2	156.8	155.8	149.7	216.4	227.8	259.3	272.9	330
331	294.0	152.7	157.3	156.4	150.2	217.1	228.5	260.0	273.7	331
332	294.9	153.2	157.9	156.9	150.7	217.8	229.2	260.8	274.6	332
333	295.8	153.7	158.4	157.4	151.2	218.4	230.0	261.6	275.4	333
334	296.7	154.2	158.9	157.9	151.7	219.1	230.7	262.4	276.2	334
335	297.6	154.7	159.4	158.4	152.3	219.8	231.4	263.2	277.0	335
336	298.5	155.2	159.9	159.0	152.8	220.5	232.1	264.0	277.9	336
337	299.3	155.8	160.5	159.5	153.3	221.1	232.8	264.8	278.7	337
338	300.2	156.3	161.0	160.0	153.8	221.8	233.5	265.6	279.5	338
339	301.1	156.8	161.5	160.5	154.3	222.5	234.2	266.4	280.4	339

TABLE 1.—MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu ₂ O)	COPPER (Cu)	DEXTROSE (d-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu ₂ O)
				0.4 gram sugar	2 grams sugar	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ ·H ₂ O	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ ·H ₂ O	
340	302.0	157.3	162.0	161.0	154.8	223.2	224.9	267.1	231.2	340
341	302.9	157.8	162.5	161.6	155.4	223.8	225.6	267.9	232.0	341
342	303.8	158.3	163.1	162.1	155.9	224.5	226.3	268.7	232.9	342
343	304.7	158.8	163.6	162.6	156.4	225.2	227.0	269.5	233.7	343
344	305.6	159.3	164.1	163.1	156.9	225.9	227.8	270.3	234.5	344
345	306.5	159.8	164.6	163.7	157.5	226.5	228.5	271.1	235.4	345
346	307.3	160.3	165.1	164.2	158.0	227.2	229.2	271.9	236.2	346
347	308.2	160.8	165.7	164.7	158.5	227.9	229.9	272.7	237.0	347
348	309.1	161.4	166.2	165.2	159.0	228.5	230.6	273.5	237.9	348
349	310.0	161.9	166.7	165.7	159.5	229.2	231.3	274.3	238.7	349
350	310.9	162.4	167.2	166.3	160.1	229.9	232.0	275.0	239.5	350
351	311.8	162.9	167.7	166.8	160.6	230.6	232.7	275.8	240.4	351
352	312.7	163.4	168.3	167.3	161.1	231.2	233.4	276.6	241.2	352
353	313.6	163.9	168.8	167.8	161.6	231.9	234.1	277.4	242.0	353
354	314.4	164.4	169.3	168.4	162.2	232.6	234.8	278.2	242.8	354
355	315.3	164.9	169.8	168.9	162.7	233.3	235.5	279.0	243.7	355
356	316.2	165.4	170.4	169.4	163.2	233.9	236.3	279.8	244.5	356
357	317.1	166.0	170.9	170.0	163.7	234.6	237.0	280.6	245.3	357
358	318.0	166.5	171.4	170.5	164.3	235.3	237.7	281.4	246.2	358
359	318.9	167.0	171.9	171.0	164.8	236.0	238.4	282.2	247.0	359
360	319.8	167.5	172.5	171.5	165.3	236.7	239.1	283.0	247.8	360
361	320.7	168.0	173.0	172.1	165.8	237.3	239.8	283.7	248.7	361
362	321.6	168.5	173.5	172.6	166.4	238.0	240.5	284.5	249.5	362
363	322.4	169.0	174.0	173.1	166.9	238.7	241.2	285.3	250.3	363
364	323.3	169.6	174.6	173.7	167.4	239.4	242.0	286.1	251.2	364
365	324.2	170.1	175.1	174.2	167.9	240.0	242.7	286.9	252.0	365
366	325.1	170.6	175.6	174.7	168.5	240.7	243.4	287.7	252.8	366
367	326.0	171.1	176.1	175.2	169.0	241.4	244.1	288.5	253.6	367
368	326.9	171.6	176.6	175.7	169.5	242.1	244.8	289.3	254.5	368
369	327.8	172.1	177.2	176.3	170.0	242.7	245.5	290.0	255.3	369
370	328.7	172.7	177.7	176.8	170.6	243.4	246.2	290.8	256.1	370
371	329.5	173.2	178.3	177.4	171.1	244.1	246.9	291.6	256.9	371
372	330.4	173.7	178.8	177.9	171.6	244.8	247.7	292.4	257.7	372
373	331.3	174.2	179.3	178.4	172.2	245.4	248.4	293.2	258.5	373
374	332.2	174.7	179.8	179.0	172.7	246.1	249.1	294.0	259.3	374
375	333.1	175.3	180.4	179.5	173.2	246.8	249.8	294.8	260.1	375
376	334.0	175.8	180.9	180.0	173.7	247.5	250.5	295.6	260.9	376
377	334.9	176.3	181.4	180.6	174.3	248.1	251.2	296.4	261.7	377
378	335.8	176.8	182.0	181.1	174.8	248.8	251.9	297.2	262.5	378
379	336.7	177.3	182.5	181.6	175.3	249.5	252.6	297.9	263.3	379
380	337.5	177.9	183.0	182.1	175.9	250.2	253.4	298.7	264.1	380
381	338.4	178.4	183.6	182.7	176.4	250.8	254.1	299.5	264.9	381
382	339.3	178.9	184.1	183.2	176.9	251.5	254.8	300.3	265.7	382
383	340.2	179.4	184.6	183.8	177.5	252.2	255.5	301.1	266.5	383
384	341.1	180.0	185.2	184.3	178.0	252.9	256.2	301.9	267.3	384
385	342.0	180.5	185.7	184.8	178.5	253.6	256.9	302.7	268.1	385
386	342.9	181.0	186.2	185.4	179.1	254.2	257.6	303.5	268.9	386
387	343.8	181.5	186.8	185.9	179.6	254.9	258.3	304.2	269.7	387
388	344.6	182.0	187.3	186.4	180.1	255.6	259.0	305.0	270.5	388
389	345.5	182.6	187.8	187.0	180.6	256.3	259.8	305.8	271.3	389
390	346.4	183.1	188.4	187.5	181.2	256.9	270.5	306.6	272.1	390
391	347.3	183.6	188.9	188.0	181.7	257.6	271.2	307.4	272.9	391
392	348.2	184.1	189.4	188.6	182.3	258.3	271.9	308.2	273.7	392
393	349.1	184.7	190.0	189.1	182.8	259.0	272.6	309.0	274.5	393
394	350.0	185.2	190.5	189.7	183.3	259.6	273.3	309.8	275.3	394

27

TABLE 1.—MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CURTIOUS OXID (C ₁₈ O)	COFFEE (Cu)	DEXTRINE (d-glucose)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CURTIOUS OXID (C ₁₈ O)
				0.4 gram sugar	2 grams sugar	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	
395	350.9	185.7	191.0	190.2	183.9	260.3	274.0	310.6	326.9	395
396	351.8	186.2	191.6	190.7	184.4	261.0	274.7	311.4	327.7	396
397	352.6	186.8	192.1	191.3	184.9	261.7	275.5	312.1	328.6	397
398	353.5	187.3	192.7	191.8	185.5	262.3	276.2	312.9	329.4	398
399	354.4	187.8	193.2	192.3	186.0	263.0	276.9	313.7	330.2	399
400	355.3	188.4	193.7	192.9	186.5	263.7	277.6	314.5	331.1	400
401	356.2	188.9	194.3	193.4	187.1	264.4	278.3	315.3	331.9	401
402	357.1	189.4	194.8	194.0	187.6	265.0	279.0	316.1	332.7	402
403	358.0	189.9	195.4	194.5	188.1	265.7	279.7	316.9	333.6	403
404	358.9	190.5	195.9	195.0	188.7	266.4	280.4	317.7	334.4	404
405	359.7	191.0	196.4	195.6	189.2	267.1	281.1	318.5	335.2	405
406	360.6	191.5	197.0	196.1	189.8	267.8	281.9	319.2	336.0	406
407	361.5	192.1	197.5	196.7	190.3	268.4	282.6	320.0	336.9	407
408	362.4	192.6	198.1	197.2	190.8	269.1	283.3	320.8	337.7	408
409	363.3	193.1	198.6	197.7	191.4	269.8	284.0	321.6	338.5	409
410	364.2	193.7	199.1	198.3	191.9	270.5	284.7	322.4	339.4	410
411	365.1	194.2	199.7	198.8	192.5	271.2	285.4	323.2	340.2	411
412	366.0	194.7	200.2	199.4	193.0	271.8	286.2	324.0	341.0	412
413	366.9	195.2	200.8	199.9	193.5	272.5	286.9	324.8	341.9	413
414	367.7	195.8	201.3	200.5	194.1	273.2	287.6	325.6	342.7	414
415	368.6	196.3	201.8	201.0	194.6	273.9	288.3	326.3	343.5	415
416	369.5	196.8	202.4	201.6	195.2	274.6	289.0	327.1	344.4	416
417	370.4	197.4	202.9	202.1	195.7	275.2	289.7	327.9	345.2	417
418	371.3	197.9	203.5	202.6	196.2	275.9	290.4	328.7	346.0	418
419	372.2	198.4	204.0	203.2	196.8	276.6	291.2	329.5	346.8	419
420	373.1	199.0	204.6	203.7	197.3	277.3	291.9	330.3	347.7	420
421	374.0	199.5	205.1	204.3	197.9	277.9	292.6	331.1	348.5	421
422	374.8	200.1	205.7	204.8	198.4	278.6	293.3	331.9	349.3	422
423	375.7	200.6	206.2	205.4	198.9	279.3	294.0	332.7	350.2	423
424	376.6	201.1	206.7	205.9	199.5	280.0	294.7	333.4	351.0	424
425	377.5	201.7	207.3	206.5	200.0	280.7	295.4	334.2	351.8	425
426	378.4	202.2	207.8	207.0	200.6	281.3	296.2	335.0	352.7	426
427	379.3	202.8	208.4	207.6	201.1	282.0	296.9	335.8	353.5	427
428	380.2	203.3	208.9	208.1	201.7	282.7	297.6	336.6	354.3	428
429	381.1	203.8	209.5	208.7	202.2	283.4	298.3	337.4	355.1	429
430	382.0	204.4	210.0	209.2	202.7	284.1	299.0	338.2	356.0	430
431	382.8	204.9	210.6	209.8	203.3	284.7	299.7	339.0	356.8	431
432	383.7	205.5	211.1	210.3	203.8	285.4	300.5	339.7	357.6	432
433	384.6	206.0	211.7	210.9	204.4	286.1	301.3	340.5	358.5	433
434	385.5	206.5	212.2	211.4	204.9	286.8	301.9	341.3	359.3	434
435	386.4	207.1	212.8	212.0	205.5	287.5	302.6	342.1	360.1	435
436	387.3	207.6	213.3	212.5	206.0	288.1	303.3	342.9	361.0	436
437	388.2	208.2	213.9	213.1	206.6	288.8	304.0	343.7	361.8	437
438	389.1	208.7	214.4	213.6	207.1	289.5	304.7	344.5	362.6	438
439	390.0	209.3	215.0	214.2	207.7	290.2	305.5	345.3	363.4	439
440	390.8	209.8	215.5	214.7	208.2	290.9	306.2	346.1	364.2	440
441	391.7	210.3	216.1	215.3	208.8	291.5	306.9	346.8	365.1	441
442	392.6	210.9	216.6	215.8	209.3	292.2	307.6	347.6	365.9	442
443	393.5	211.4	217.2	216.4	209.9	292.9	308.3	348.4	366.8	443
444	394.4	212.0	217.8	216.9	210.4	293.6	309.0	349.2	367.6	444
445	395.3	212.5	218.3	217.5	211.0	294.2	309.7	350.0	368.4	445
446	396.2	213.1	218.9	218.0	211.5	294.9	310.5	350.8	369.3	446
447	397.1	213.6	219.4	218.6	212.1	295.6	311.2	351.6	370.1	447
448	397.9	214.1	220.0	219.1	212.6	296.3	311.9	352.4	370.9	448
449	398.8	214.7	220.5	219.7	213.2	297.0	312.6	353.2	371.7	449

27

TABLE 1.—MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu ₂ O)	COPPER (Cu)	DEXTROSE (β-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu ₂ O)
				0.4 gram sugar	3 grams sugar	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	C ₁₂ H ₂₂ O ₁₁	C ₁₂ H ₂₂ O ₁₁ H ₂ O	
450	399.7	215.2	221.1	220.2	213.7	297.6	313.3	353.9	372.6	450
451	400.6	215.8	221.6	220.8	214.3	298.3	314.0	354.7	373.4	451
452	401.5	216.3	222.2	221.4	214.8	299.0	314.7	355.5	374.2	452
453	402.4	216.9	222.8	221.9	215.4	299.7	315.5	356.3	375.1	453
454	403.3	217.4	223.3	222.5	215.9	300.4	316.2	357.1	375.9	454
455	404.2	218.0	223.9	223.0	216.5	301.1	316.9	357.9	376.7	455
456	405.1	218.5	224.4	223.6	217.0	301.7	317.6	358.7	377.6	456
457	405.9	219.1	225.0	224.1	217.6	302.4	318.3	359.5	378.4	457
458	406.8	219.6	225.5	224.7	218.1	303.1	319.0	360.3	379.2	458
459	407.7	220.2	226.1	225.3	218.7	303.8	319.8	361.0	380.0	459
460	408.6	220.7	226.7	225.8	219.2	304.5	320.5	361.8	380.9	460
461	409.5	221.3	227.2	226.4	219.8	305.1	321.2	362.6	381.7	461
462	410.4	221.8	227.8	226.9	220.3	305.8	321.9	363.4	382.5	462
463	411.3	222.4	228.3	227.5	220.9	306.5	322.6	364.2	383.4	463
464	412.2	222.9	228.9	228.1	221.4	307.2	323.4	365.0	384.2	464
465	413.0	223.5	229.5	228.6	222.0	307.9	324.1	365.8	385.0	465
466	413.9	224.0	230.0	229.2	222.5	308.6	324.8	366.6	385.9	466
467	414.8	224.6	230.6	229.7	223.1	309.2	325.5	367.3	386.7	467
468	415.7	225.1	231.2	230.3	223.7	309.9	326.2	368.1	387.5	468
469	416.6	225.7	231.7	230.9	224.2	310.6	326.9	368.9	388.3	469
470	417.5	226.2	232.3	231.4	224.8	311.3	327.7	369.7	389.2	470
471	418.4	226.8	232.8	232.0	225.3	312.0	328.4	370.5	390.0	471
472	419.3	227.4	233.4	232.5	225.9	312.6	329.1	371.3	390.8	472
473	420.2	227.9	234.0	233.1	226.4	313.3	329.8	372.1	391.7	473
474	421.0	228.5	234.5	233.7	227.0	314.0	330.5	372.9	392.5	474
475	421.9	229.0	235.1	234.2	227.6	314.7	331.3	373.7	393.3	475
476	422.8	229.6	235.7	234.8	228.1	315.4	332.0	374.4	394.2	476
477	423.7	230.1	236.2	235.4	228.7	316.1	332.7	375.2	395.0	477
478	424.6	230.7	236.8	235.9	229.2	316.7	333.4	376.0	395.8	478
479	425.5	231.3	237.4	236.5	229.8	317.4	334.1	376.8	396.6	479
480	426.4	231.8	237.9	237.1	230.3	318.1	334.8	377.6	397.5	480
481	427.3	232.4	238.5	237.6	230.9	318.8	335.6	378.4	398.3	481
482	428.1	232.9	239.1	238.2	231.5	319.5	336.3	379.2	399.1	482
483	429.0	233.5	239.6	238.8	232.0	320.1	337.0	380.0	400.0	483
484	429.9	234.1	240.2	239.3	232.6	320.8	337.7	380.7	400.8	484
485	430.8	234.6	240.8	239.9	233.2	321.5	338.4	381.5	401.6	485
486	431.7	235.2	241.4	240.5	233.7	322.2	339.1	382.3	402.4	486
487	432.6	235.7	241.9	241.0	234.3	322.9	339.9	383.1	403.3	487
488	433.5	236.3	242.5	241.6	234.8	323.6	340.6	383.9	404.1	488
489	434.4	236.9	243.1	242.2	235.4	324.2	341.3	384.7	404.9	489
490	435.3	237.4	243.6	242.7	236.0	324.9	342.0	385.5	405.8	490

II. A. H. Low Volumetric Method, Modified.⁴—Tentative.

28

REAGENT.

Standard thiosulphate solution.—Prepare a solution of sodium thiosulphate containing 19 grams of pure crystals in 1 liter. Weigh accurately about 0.2 gram of pure copper foil and place in a flask of 250 cc. capacity. Dissolve by warming with 5 cc. of a mixture of equal volumes of strong nitric acid and water. Dilute to 50 cc., boil to expel the red fumes, add 5 cc. of strong bromin water, and boil

until the bromin is completely driven off. Remove from the heat and add a slight excess of strong ammonium hydroxid (about 7 cc. is required). Again boil until the excess of ammonia is expelled, as shown by a change of color of the liquid, and a partial precipitation. Then add a slight excess of strong acetic acid (3 or 4 cc. of 80% acid) and boil for a minute. Cool to room temperature and add 10 cc. of 30% potassium iodid solution. Titrate at once with the thiosulphate solution until the brown tinge has become weak, then add sufficient starch indicator (VII, 3 (a)) to produce a marked blue coloration. Continue the titration cautiously until the color due to free iodin has entirely vanished. The blue color changes toward the end to a faint lilac. If at this point the thiosulphate be added drop by drop and a little time allowed for complete reaction after each addition, there is no difficulty in determining the end point within a single drop. One cc. of the thiosulphate solution will be found to correspond to about 0.005 gram of copper.

29

DETERMINATION.

After washing the precipitated cuprous oxid, cover the Gooch with a watch glass and dissolve the oxid by means of 5 cc. of warm nitric acid (1 to 1) poured under the watch glass with a pipette. Catch the filtrate in a 250 cc. flask, wash the watch glass and Gooch free of copper, using about 50 cc. of water. Boil to expel red fumes, add 5 cc. of bromin water, boil off the bromin, and proceed exactly as in 28.

30

III. Volumetric Permanganate Method.—Tentative.

Filter and wash the cuprous oxid as directed under 25. Transfer the asbestos film to the beaker, add about 30 cc. of hot water, and beat the precipitate and asbestos thoroughly. Rinse the crucible with 50 cc. of a hot saturated solution of ferric sulphate in 20% sulphuric acid, receiving the rinsings in the beaker containing the precipitate. After the cuprous oxid is dissolved, wash the solution into a large Erlenmeyer flask and immediately titrate with a standard solution of potassium permanganate, 1 cc. of which should be equivalent to 0.010 gram of copper. Standardize this solution by making 6 or more determinations with the same sugar solution, titrating one half of the precipitates obtained, and determining the copper in the others by electrolysis. The average weight of copper obtained by electrolysis, divided by the average number of cc. of permanganate solution required for the titrations, gives the weight of copper equivalent to 1 cc. of the standard permanganate solution. A solution standardized with iron or oxalic acid will give too low a result.

31

IV. Electrolytic Deposition from Sulphuric Acid Solution.—Tentative.

Filter the cuprous oxid in a Gooch, wash the beaker and the precipitate thoroughly with hot water without transferring the precipitate to the filter. Wash the asbestos film and the adhering cuprous oxid into the beaker by means of hot dilute nitric acid. After the copper is all in solution, refilter through a thin film of asbestos in a Gooch and wash thoroughly with hot water. Add 10 cc. of sulphuric acid (1 to 4), and evaporate the filtrate on the steam bath until the copper salt has largely crystallized. Heat carefully on a hot plate or over asbestos until the evolution of white fumes shows that the excess of nitric acid is removed. Add 8-10 drops of nitric acid (sp. gr. 1.42) and rinse into a 100-125 cc. platinum dish. Deposit the copper by

electrolysis. Wash thoroughly with water, then break the current, wash with alcohol and ether successively, dry at about 50°C., and weigh. If preferred, the electrolysis can be conducted in a beaker, the copper being deposited upon a weighed platinum electrode.

32 *V. Electrolytic Deposition from Sulphuric and Nitric Acid Solution.—Tentative.*

Filter and wash as directed under 31. Transfer the asbestos film from the crucible to the beaker by means of a glass rod and rinse the crucible with about 30 cc. of a boiling mixture of dilute sulphuric and nitric acids, containing 65 cc. of sulphuric acid (sp. gr. 1.84) and 50 cc. of nitric acid (sp. gr. 1.42) per liter. Heat and agitate until solution is complete; filter and electrolyze as under 31.

33 *VI. Electrolytic Deposition from Nitric Acid Solution.—Tentative.*

Filter and wash as directed under 31. Transfer the asbestos film and adhering oxid to the beaker. Dissolve the oxid still remaining in the crucible by means of 2 cc. of nitric acid (sp. gr. 1.42), adding it with a pipette and receiving the solution in the beaker containing the asbestos film. Rinse the crucible with a jet of water, allowing the rinsings to flow into the beaker. Heat the contents of the beaker until the copper is all in solution, filter, dilute the filtrate to a volume of 100 cc. or more, and electrolyze. When a nitrate solution is electrolyzed, the first washing of the deposit should be made with water acidulated with sulphuric acid, in order to remove all the nitric acid before the current is interrupted.

34 *VII. Reduction in Hydrogen.—Tentative.*

Deposit an asbestos film on a perforated platinum disc or cone contained in a hard glass filtering tube, wash free from loose fibers, dry and weigh. Through this tube, previously moistened, filter the cuprous oxid immediately, using suction. Transfer the cuprous oxid to the tube through a removable funnel, and wash thoroughly with hot water, alcohol and ether successively. After drying, connect the tube with a supply of dry hydrogen, heat gently until the cuprous oxid is completely reduced to metallic copper, cool in the current of hydrogen, and weigh. If preferred, a Gooch crucible may be used for the filtration.

Herzfeld Gravimetric Method.—Tentative.

Method I.

(For materials containing 1.5% or less of invert sugar and 98.5% or more of sucrose.)

35 REAGENTS.

The reagents and solutions used are described under 24.

36 DETERMINATION.

Prepare the solution of the material to be examined so as to contain 20 grams in 100 cc., free from suspended impurities by filtration and from soluble impurities by neutral lead acetate, removing the excess of lead by means of sodium carbonate. Place 50 cc. of the reagent and 50 cc. of the sugar solution in a 250 cc. beaker. Heat this mixture at such a rate that approximately 4 minutes are required to bring it to the boiling point, and boil for exactly 2 minutes. Add 100 cc. of cold, recently boiled, water. Filter immediately through asbestos, and determine the copper by one of the methods under 26, 29-34, respectively. Obtain the corresponding percentage of invert sugar from 37.

37

TABLE 2.—HERZFELD'S TABLE.¹

For the determination of invert sugar in materials containing 1.5%, or less, of invert sugar and 98.5%, or more, of sucrose.

COPPER REDUCED BY 10 GRAMS OF MATERIAL	INVERT SUGAR	COPPER REDUCED BY 10 GRAMS OF MATERIAL	INVERT SUGAR	COPPER REDUCED BY 10 GRAMS OF MATERIAL	INVERT SUGAR
mg.	per cent	mg.	per cent	mg.	per cent
50	0.05	140	0.51	230	1.02
55	0.07	145	0.53	235	1.05
60	0.09	150	0.56	240	1.07
65	0.11	155	0.59	245	1.10
70	0.14	160	0.62	250	1.13
75	0.16	165	0.65	255	1.16
80	0.19	170	0.68	260	1.18
85	0.21	175	0.71	265	1.21
90	0.24	180	0.74	270	1.24
95	0.27	185	0.76	275	1.27
100	0.30	190	0.79	280	1.30
105	0.32	195	0.82	285	1.33
110	0.35	200	0.85	290	1.36
115	0.38	205	0.88	295	1.38
120	0.40	210	0.90	300	1.41
125	0.43	215	0.93	305	1.44
130	0.45	220	0.96	310	1.47
135	0.48	225	0.99	315	1.50

Method II.

(For materials containing 1.5% or more of invert sugar and 98.5% or less of sucrose.)

38

REAGENTS.

Same as described under 24.

39

DETERMINATION.

Prepare a solution of the material to be examined in such a manner that it contains 20 grams in 100 cc. after clarification and removal of the excess of lead. Prepare a series of solutions in large test tubes by adding 1, 2, 3, 4, and 5 cc. of this solution to each tube successively. Add 5 cc. of the reagent to each, heat to boiling, boil 2 minutes, and filter. Note the volume of sugar solution which gives the filtrate lightest in tint, but still distinctly blue. Place 20 times this volume of the sugar solution in a 100 cc. flask, dilute to the mark, and mix well. Use 50 cc. of the solution for the determination, which is conducted as described under 36. For the calculation of the result use the following formulas and table of factors of Meissl and Hiller:

Let Cu = the weight of copper obtained;

P = the polarization of the sample;

W = the weight of the sample in the 50 cc. of the solution used for the determination;

F = the factor obtained from the table for the conversion of copper to invert sugar;

Then $\frac{\text{Cu}}{2} = Z$, approximate weight of invert sugar;

$Z \times \frac{100}{W} = Y$, approximate per cent of invert sugar;

$$\frac{100 P}{P + Y} = R, \text{ approximate per cent of sucrose in mixture of sugars;}$$

$$100 - R = I, \text{ approximate per cent of invert sugar;}$$

$$\frac{CuF}{W} = \text{per cent of invert sugar.}$$

The factor F for calculating copper to invert sugar is then found from 40.

40

TABLE 3.

Meissl and Hiller's⁴ factors for determinations in materials in which, of the total sugars present, 1.5%, or more, is invert sugar, and 98.5%, or less, is sucrose.

RATIO OF SUCROSE TO INVERT SUGAR = R:I.	APPROXIMATE ABSOLUTE WEIGHT OF INVERT SUGAR (Z)						
	200 milligrams	175 milligrams	150 milligrams	125 milligrams	100 milligrams	75 milligrams	50 milligrams
	per cent	per cent	per cent	per cent	per cent	per cent	per cent
0:100	56.4	55.4	54.5	53.8	53.2	53.0	53.0
10:90	56.3	55.3	54.4	53.8	53.2	52.9	52.9
20:80	56.2	55.2	54.3	53.7	53.2	52.7	52.7
30:70	56.1	55.1	54.2	53.7	53.2	52.6	52.6
40:60	55.9	55.0	54.1	53.6	53.1	52.5	52.4
50:50	55.7	54.9	54.0	53.5	53.1	52.3	52.2
60:40	55.6	54.7	53.8	53.2	52.8	52.1	51.9
70:30	55.5	54.5	53.5	52.9	52.5	51.9	51.6
80:20	55.4	54.3	53.3	52.7	52.2	51.7	51.3
90:10	54.6	53.6	53.1	52.6	52.1	51.6	51.2
91:9	54.1	53.6	52.6	52.1	51.6	51.2	50.7
92:8	53.6	53.1	52.1	51.6	51.2	50.7	50.3
93:7	53.6	53.1	52.1	51.2	50.7	50.3	49.8
94:6	53.1	52.6	51.6	50.7	50.3	49.8	48.9
95:5	52.6	52.1	51.2	50.3	49.4	48.9	48.5
96:4	52.1	51.2	50.7	49.8	48.9	47.7	46.9
97:3	50.7	50.3	49.8	48.9	47.7	46.2	45.1
98:2	49.9	48.9	48.5	47.3	45.8	43.3	40.0
99:1	47.7	47.3	46.5	45.1	43.3	41.2	38.1

Example: The polarization of a sugar is 86.4, and 50 cc. of solution containing 3.256 grams of sample gave 0.290 gram of copper.

$$\frac{Cu}{2} = \frac{0.290}{2} = 0.145 = Z$$

$$\frac{Z \times 100}{W} = 0.145 \times \frac{100}{3.256} = 4.45 = Y$$

$$100 - R = 100 - 95.1 = I = 4.9$$

$$R:I = 95.1:4.9$$

By consulting the table it will be seen that the vertical column headed 150 is nearest to Z, 145, and the horizontal column headed 95:5 is nearest to the ratio of R to I, 95.1:4.9. Where these columns meet, we find the factor 51.2 which enters into the final calculation:

$$\frac{CuF}{W} = \frac{0.290 \times 51.2}{3.256} = 4.56 \text{ per cent of invert sugar.}$$

In case there is no sucrose present, the following table may be used instead of the factors given in 40.

TABLE 4.—MEISSL'S TABLE.⁷
For the determination of invert sugar alone.

[According to Wein.]

[Expressed in milligrams.]

COPPER	INVERT SUGAR	COPPER	INVERT SUGAR	COPPER	INVERT SUGAR	COPPER	INVERT SUGAR
90	46.9	135	70.8	180	95.2	225	120.4
91	47.4	136	71.3	181	95.7	226	120.9
92	47.9	137	71.9	182	96.2	227	121.5
93	48.4	138	72.4	183	96.8	228	122.1
94	48.9	139	72.9	184	97.3	229	122.6
95	49.5	140	73.5	185	97.8	230	123.2
96	50.0	141	74.0	186	98.4	231	123.8
97	50.5	142	74.5	187	99.0	232	124.3
98	51.1	143	75.1	188	99.5	233	124.9
99	51.6	144	75.6	189	100.1	234	125.5
100	52.1	145	76.1	190	100.6	235	126.0
101	52.7	146	76.7	191	101.2	236	126.6
102	53.2	147	77.2	192	101.7	237	127.2
103	53.7	148	77.8	193	102.3	238	127.8
104	54.3	149	78.3	194	102.9	239	128.3
105	54.8	150	78.9	195	103.4	240	128.9
106	55.3	151	79.4	196	104.0	241	129.5
107	55.9	152	80.0	197	104.6	242	130.0
108	56.4	153	80.5	198	105.1	243	130.6
109	56.9	154	81.0	199	105.7	244	131.2
110	57.5	155	81.6	200	106.3	245	131.8
111	58.0	156	82.1	201	106.8	246	132.3
112	58.5	157	82.7	202	107.4	247	132.9
113	59.1	158	83.2	203	107.9	248	133.5
114	59.6	159	83.8	204	108.5	249	134.1
115	60.1	160	84.3	205	109.1	250	134.6
116	60.7	161	84.8	206	109.6	251	135.2
117	61.2	162	85.4	207	110.2	252	135.8
118	61.7	163	85.9	208	110.8	253	136.3
119	62.3	164	86.5	209	111.3	254	136.9
120	62.8	165	87.0	210	111.9	255	137.5
121	63.3	166	87.6	211	112.5	256	138.1
122	63.9	167	88.1	212	113.0	257	138.6
123	64.4	168	88.6	213	113.6	258	139.2
124	64.9	169	89.2	214	114.2	259	139.8
125	65.5	170	89.7	215	114.7	260	140.4
126	66.0	171	90.3	216	115.3	261	140.9
127	66.5	172	90.8	217	115.8	262	141.5
128	67.1	173	91.4	218	116.4	263	142.1
129	67.6	174	91.9	219	117.0	264	142.7
130	68.1	175	92.4	220	117.5	265	143.2
131	68.7	176	93.0	221	118.1	266	143.8
132	69.2	177	93.5	222	118.7	267	144.4
133	69.7	178	94.1	223	119.2	268	144.9
134	70.3	179	94.6	224	119.8	269	145.5

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TABLE 4.—MEISSL'S TABLE.—Continued.

[Expressed in milligrams.]

COFFER	INVERT SUGAR	COFFER	INVERT SUGAR	COFFER	INVERT SUGAR	COFFER	INVERT SUGAR
270	146.1	310	169.7	350	193.8	390	218.7
271	146.7	311	170.3	351	194.4	391	219.3
272	147.2	312	170.9	352	195.0	392	219.9
273	147.8	313	171.5	353	195.6	393	220.5
274	148.4	314	172.1	354	196.2	394	221.2
275	149.0	315	172.7	355	196.8	395	221.8
276	149.5	316	173.3	356	197.4	396	222.4
277	150.1	317	173.9	357	198.0	397	223.1
278	150.7	318	174.5	358	198.6	398	223.7
279	151.3	319	175.1	359	199.2	399	224.3
280	151.9	320	175.6	360	199.8	400	224.9
281	152.5	321	176.2	361	200.4	401	225.7
282	153.1	322	176.8	362	201.1	402	226.4
283	153.7	323	177.4	363	201.7	403	227.1
284	154.3	324	178.0	364	202.3	404	227.8
285	154.9	325	178.6	365	203.0	405	228.6
286	155.5	326	179.2	366	203.6	406	229.3
287	156.1	327	179.8	367	204.2	407	230.0
288	156.7	328	180.4	368	204.8	408	230.7
289	157.2	329	181.0	369	205.5	409	231.4
290	157.8	330	181.6	370	206.1	410	232.1
291	158.4	331	182.2	371	206.7	411	232.8
292	159.0	332	182.8	372	207.3	412	233.5
293	159.6	333	183.5	373	208.0	413	234.3
294	160.2	334	184.1	374	208.6	414	235.0
295	160.8	335	184.7	375	209.2	415	235.7
296	161.4	336	185.4	376	209.9	416	236.4
297	162.0	337	186.0	377	210.5	417	237.1
298	162.6	338	186.6	378	211.1	418	237.8
299	163.2	339	187.2	379	211.7	419	238.5
300	163.8	340	187.8	380	212.4	420	239.2
301	164.4	341	188.4	381	213.0	421	239.9
302	165.0	342	189.0	382	213.6	422	240.6
303	165.6	343	189.6	383	214.3	423	241.3
304	166.2	344	190.2	384	214.9	424	242.0
305	166.8	345	190.8	385	215.5	425	242.7
306	167.3	346	191.4	386	216.1	426	243.4
307	167.9	347	192.0	387	216.8	427	244.1
308	168.5	348	192.6	388	217.4	428	244.9
309	169.1	349	193.2	389	218.0	429	245.6
						430	246.3

MALTOSE.

42

General Gravimetric Method.—Tentative.

Proceed as directed under 25 and obtain, from 27, the weight of maltose equivalent to the weight of copper reduced.

Wein Method.—Tentative.

43

REAGENTS.

The reagents and solutions used are described under 24.

44

DETERMINATION.

Place 50 cc. of the reagent in a beaker and heat to the boiling point. When boiling briskly, add 25 cc. of the maltose solution containing not more than 0.250 gram of maltose and boil for 4 minutes. Filter immediately through asbestos and determine, by one of the methods given under 26, 29–34 respectively, the amount of copper reduced.

Obtain, from 45, the weight of maltose equivalent to the weight of copper found.

45

TABLE 5.

*For the determination of maltose.**[According to Wein.*]**[Expressed in milligrams.]*

COPPER	CU- PROUS OXID	MAL- TOSE	COPPER	CU- PROUS OXID	MAL- TOSE	COPPER	CU- PROUS OXID	MAL- TOSE	COPPER	CU- PROUS OXID	MAL- TOSE
31	34.9	26.1	71	79.9	61.0	111	125.0	96.4	151	170.0	132.3
32	36.0	27.0	72	81.1	61.8	112	126.1	97.3	152	171.1	133.2
33	37.2	27.9	73	82.2	62.7	113	127.2	98.1	153	172.2	134.1
34	38.3	28.7	74	83.3	63.6	114	128.3	99.0	154	173.4	135.0
35	39.4	29.6	75	84.4	64.5	115	129.6	99.9	155	174.5	135.9
36	40.5	30.5	76	85.6	65.4	116	130.6	100.8	156	175.6	136.8
37	41.7	31.3	77	86.7	66.2	117	131.7	101.7	157	176.8	137.7
38	42.8	32.2	78	87.8	67.1	118	132.8	102.6	158	177.9	138.6
39	43.9	33.1	79	88.9	68.0	119	134.0	103.5	159	179.0	139.5
40	45.0	33.9	80	90.1	68.9	120	135.1	104.4	160	180.1	140.4
41	46.2	34.8	81	91.2	69.7	121	136.2	105.3	161	181.3	141.3
42	47.3	35.7	82	92.3	70.6	122	137.4	106.2	162	182.4	142.2
43	48.4	36.5	83	93.4	71.5	123	138.5	107.1	163	183.5	143.1
44	49.5	37.4	84	94.6	72.4	124	139.6	108.0	164	184.6	144.0
45	50.7	38.3	85	95.7	73.2	125	140.7	108.9	165	185.8	144.9
46	51.8	39.1	86	96.8	74.1	126	141.9	109.8	166	186.9	145.8
47	52.9	40.0	87	97.9	75.0	127	143.0	110.7	167	188.0	146.7
48	54.0	40.9	88	99.1	75.9	128	144.1	111.6	168	189.1	147.6
49	55.2	41.8	89	100.2	76.8	129	145.2	112.5	169	190.3	148.5
50	56.3	42.6	90	101.3	77.7	130	146.4	113.4	170	191.4	149.4
51	57.4	43.5	91	102.4	78.6	131	147.5	114.3	171	192.5	150.3
52	58.5	44.4	92	103.6	79.5	132	148.6	115.2	172	193.6	151.2
53	59.7	45.2	93	104.7	80.3	133	149.7	116.1	173	194.8	152.0
54	60.8	46.1	94	105.8	81.2	134	150.9	117.0	174	195.9	152.9
55	61.9	47.0	95	107.0	82.1	135	152.0	117.9	175	197.0	153.8
56	63.0	47.8	96	108.1	83.0	136	153.1	118.8	176	198.1	154.7
57	64.2	48.7	97	109.2	83.9	137	154.2	119.7	177	199.3	155.6
58	65.3	49.6	98	110.3	84.8	138	155.4	120.6	178	200.4	156.5
59	66.4	50.4	99	111.5	85.7	139	156.5	121.5	179	201.5	157.4
60	67.6	51.3	100	112.6	86.6	140	157.6	122.4	180	202.6	158.3
61	68.7	52.2	101	113.7	87.5	141	158.7	123.3	181	203.8	159.2
62	69.8	53.1	102	114.8	88.4	142	159.9	124.2	182	204.9	160.1
63	70.9	53.9	103	116.0	89.2	143	161.0	125.1	183	206.0	160.9
64	72.1	54.8	104	117.1	90.1	144	162.1	126.0	184	207.1	161.8
65	73.2	55.7	105	118.2	91.0	145	163.2	126.9	185	208.3	162.7
66	74.3	56.6	106	119.3	91.9	146	164.4	127.8	186	209.4	163.6
67	75.4	57.4	107	120.5	92.8	147	165.5	128.7	187	210.5	164.5
68	76.6	58.3	108	121.6	93.7	148	166.6	129.6	188	211.7	165.4
69	77.7	59.2	109	122.7	94.6	149	167.7	130.5	189	212.8	166.3
70	78.8	60.1	110	123.8	95.5	150	168.9	131.4	190	213.9	167.2

45

TABLE 5.—Continued.
For the determination of maltose.
[Expressed in milligrams.]

COPPER	CU- PROUS OXID	MAL- TOSE	COPPER	CU- PROUS OXID	MAL- TOSE	COPPER	CU- PROUS OXID	MAL- TOSE	COPPER	CU- PROUS OXID	MAL- TOSE
191	215.0	168.1	221	248.7	194.8	251	282.6	221.7	281	316.4	248.7
192	216.2	169.0	222	249.9	195.7	252	283.7	222.6	282	317.5	249.6
193	217.3	169.8	223	251.0	196.6	253	284.8	223.5	283	318.6	250.4
194	218.4	170.7	224	252.4	197.5	254	286.0	224.4	284	319.7	251.2
195	219.5	171.6	225	253.3	198.4	255	287.1	225.3	285	320.9	252.2
196	220.7	172.5	226	254.4	199.3	256	288.2	226.2	286	322.0	253.1
197	221.8	173.4	227	255.6	200.2	257	289.3	227.1	287	323.1	254.0
198	222.9	174.3	228	256.7	201.1	258	290.5	228.0	288	324.2	254.9
199	224.0	175.2	229	257.8	202.0	259	291.6	228.9	289	325.4	255.8
200	225.2	176.1	230	258.9	202.9	260	292.7	229.8	290	326.5	256.6
201	226.3	177.0	231	260.1	203.8	261	293.8	230.7	291	327.4	257.5
202	227.4	177.9	232	261.2	204.7	262	295.0	231.6	292	328.7	258.4
203	228.5	178.7	233	262.3	205.6	263	296.1	232.5	293	329.9	259.2
204	229.7	179.6	234	263.4	206.5	264	297.2	233.4	294	331.0	260.2
205	230.8	180.5	235	264.6	207.4	265	298.3	234.3	295	332.1	261.1
206	231.9	181.4	236	265.7	208.3	266	299.5	235.2	296	333.2	262.0
207	233.0	182.3	237	266.8	209.1	267	300.6	236.1	297	334.4	262.8
208	234.2	183.2	238	268.0	210.0	268	301.7	237.0	298	335.5	263.7
209	235.3	184.1	239	269.1	210.9	269	302.8	237.9	299	336.6	264.6
210	236.4	185.0	240	270.2	211.8	270	304.0	238.8	300	337.8	265.5
211	237.6	185.9	241	271.3	212.7	271	305.1	239.7			
212	238.7	186.8	242	272.5	213.6	272	306.2	240.6			
213	239.8	187.7	243	273.6	214.5	273	307.3	241.5			
214	240.9	188.6	244	274.7	215.4	274	308.5	242.4			
215	242.1	189.5	245	275.8	216.3	275	309.6	243.3			
216	243.2	190.4	246	277.0	217.2	276	310.7	244.2			
217	244.3	191.2	247	278.1	218.1	277	311.9	245.1			
218	245.4	192.1	248	279.2	219.0	278	313.0	246.0			
219	246.6	193.0	249	280.3	219.9	279	314.1	246.9			
220	247.7	193.9	250	281.5	220.8	280	315.2	247.8			

LACTOSE.

46

General Gravimetric Method.—Tentative.

Proceed as directed under 25 and obtain, from 27, the weight of lactose equivalent to the weight of copper reduced.

Soxhlet-Wein Method.—Official.

47

REAGENTS.

The reagents and solutions used are described under 24.

48

DETERMINATION.

Place 50 cc. of the reagent in a beaker and heat to the boiling point. When boiling briskly, add 100 cc. of the lactose solution containing not more than 0.300 gram of lactose and boil for 6 minutes. Filter immediately through asbestos and determine by one of the methods given under 26, 29-34 inclusive, the amount of copper reduced. Obtain, from 49, the weight of lactose equivalent to the weight of copper found.

TABLE 6.

For the determination of lactose (Soxhlet-Wein).*

[Expressed in milligrams.]

COFFER	LACTOSE	COFFER	LACTOSE	COFFER	LACTOSE	COFFER	LACTOSE	COFFER	LACTOSE
100	71.6	160	118.4	220	161.9	280	208.3	340	255.7
101	72.4	161	117.1	221	162.7	281	209.1	341	256.6
102	73.1	162	117.9	222	163.4	282	209.9	342	257.4
103	73.8	163	118.6	223	164.2	283	210.7	343	258.2
104	74.6	164	119.4	224	164.9	284	211.5	344	259.0
105	75.3	165	120.2	225	165.7	285	212.3	345	259.8
106	76.1	166	120.9	226	166.4	286	213.1	346	260.6
107	76.8	167	121.7	227	167.2	287	213.9	347	261.4
108	77.6	168	122.4	228	167.9	288	214.7	348	262.3
109	78.3	169	123.2	229	168.6	289	215.5	349	263.1
110	79.0	170	123.9	230	169.4	290	216.3	350	263.9
111	79.8	171	124.7	231	170.1	291	217.1	351	264.7
112	80.5	172	125.5	232	170.9	292	217.9	352	265.5
113	81.3	173	126.2	233	171.6	293	218.7	353	266.3
114	82.0	174	127.0	234	172.4	294	219.5	354	267.2
115	82.7	175	127.8	235	173.1	295	220.3	355	268.0
116	83.5	176	128.5	236	173.9	296	221.1	356	268.8
117	84.2	177	129.3	237	174.6	297	221.9	357	269.6
118	85.0	178	130.1	238	175.4	298	222.7	358	270.4
119	85.7	179	130.8	239	176.2	299	223.5	359	271.2
120	86.4	180	131.6	240	176.9	300	224.4	360	272.1
121	87.2	181	132.4	241	177.7	301	225.2	361	272.9
122	87.9	182	133.1	242	178.5	302	225.9	362	273.7
123	88.7	183	133.9	243	179.3	303	226.7	363	274.5
124	89.4	184	134.7	244	180.1	304	227.5	364	275.3
125	90.1	185	135.4	245	180.8	305	228.3	365	276.2
126	90.9	186	136.2	246	181.6	306	229.1	366	277.1
127	91.6	187	137.0	247	182.4	307	229.8	367	277.9
128	92.4	188	137.7	248	183.2	308	230.6	368	278.8
129	93.1	189	138.5	249	184.0	309	231.4	369	279.6
130	93.8	190	139.3	250	184.8	310	232.2	370	280.5
131	94.6	191	140.0	251	185.5	311	232.9	371	281.4
132	95.3	192	140.8	252	186.3	312	233.7	372	282.2
133	96.1	193	141.6	253	187.1	313	234.5	373	283.1
134	96.9	194	142.3	254	187.9	314	235.3	374	283.9
135	97.6	195	143.1	255	188.7	315	236.1	375	284.8
136	98.3	196	143.9	256	189.4	316	236.8	376	285.7
137	99.1	197	144.6	257	190.2	317	237.6	377	286.5
138	99.8	198	145.4	258	191.0	318	238.4	378	287.4
139	100.5	199	146.2	259	191.8	319	239.2	379	288.2
140	101.3	200	146.9	260	192.5	320	240.0	380	289.1
141	102.0	201	147.7	261	193.3	321	240.7	381	289.9
142	102.8	202	148.5	262	194.1	322	241.5	382	290.8
143	103.5	203	149.2	263	194.9	323	242.3	383	291.7
144	104.3	204	150.0	264	195.7	324	243.1	384	292.5
145	105.1	205	150.7	265	196.4	325	243.9	385	293.4
146	105.8	206	151.5	266	197.2	326	244.6	386	294.2
147	106.6	207	152.2	267	198.0	327	245.4	387	295.1
148	107.3	208	153.0	268	198.8	328	246.2	388	296.0
149	108.1	209	153.7	269	199.5	329	247.0	389	296.8
150	108.8	210	154.5	270	200.3	330	247.7	390	297.7
151	109.6	211	155.2	271	201.1	331	248.5	391	298.5
152	110.3	212	156.0	272	201.9	332	249.2	392	299.4
153	111.1	213	156.7	273	202.7	333	250.0	393	300.3
154	111.9	214	157.5	274	203.5	334	250.8	394	301.1
155	112.6	215	158.2	275	204.3	335	251.6	395	302.0
156	113.4	216	159.0	276	205.1	336	252.5	396	302.8
157	114.1	217	159.7	277	205.9	337	253.3	397	303.7
158	114.9	218	160.4	278	206.7	338	254.1	398	304.6
159	115.6	219	161.2	279	207.5	339	254.9	399	305.4
								400	306.3

DEXTROSE.

50 *Approximate Volumetric Method for Rapid Work.—Tentative.*

Proceed as directed under 21. Standardize the reagent against pure dextrose.

51 *Soxhlet Method.—Tentative.*

Proceed as directed under 23. Under these conditions 100 cc. of the reagent require 0.475 gram of anhydrous dextrose for complete reduction and the formula becomes $\frac{100 \times 0.475}{VW} = \text{per cent of dextrose.}$

52 *General Gravimetric Method.—Tentative.*

Proceed as directed under 25 and obtain, from 27, the weight of dextrose equivalent to the weight of copper reduced.

*Allihn Gravimetric Method.—Tentative.***53** REAGENT.

Allihn's Modification of Fehling's Solution.—Prepare by mixing, immediately before use, equal volumes of (a) and (b).

(a) *Copper sulphate solution.*—Dissolve 34.639 grams of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water and dilute to 500 cc.

(b) *Alkaline tartrate solution.*—Dissolve 173 grams of Rochelle salts and 125 grams of potassium hydroxid in water and dilute to 500 cc.

54 DETERMINATION.

Place 30 cc. of the copper sulphate solution, 30 cc. of the alkaline tartrate solution, and 60 cc. of water in a beaker and heat to boiling. Add 25 cc. of the solution of the material to be examined, prepared so as not to contain more than 0.25 gram of dextrose, and boil for exactly 2 minutes, keeping the beaker covered. Filter immediately through asbestos, and obtain the weight of copper by one of the methods given under 26, 29–34 inclusive. The corresponding weight of dextrose is found in 55.

TABLE 7.—ALLIHN'S TABLE.¹⁰*For the determination of dextrose.*

[Expressed in milligrams.]

COFFER	CU- PROUS OXID	DEX- TROSE	COFFER	CU- PROUS OXID	DEX- TROSE	COFFER	CU- PROUS OXID	DEX- TROSE	COFFER	CU- PROUS OXID	DEX- TROSE
11	12.4	6.6	71	79.9	36.3	131	147.5	66.7	191	215.0	97.8
12	13.5	7.1	72	81.1	36.8	132	148.6	67.2	192	216.2	98.4
13	14.6	7.6	73	82.2	37.3	133	149.7	67.7	193	217.3	98.9
14	15.8	8.1	74	83.3	37.8	134	150.9	68.2	194	218.4	99.4
15	16.9	8.6	75	84.4	38.3	135	152.0	68.8	195	219.5	100.0
16	18.0	9.0	76	85.6	38.8	136	153.1	69.3	196	220.7	100.5
17	19.1	9.5	77	86.7	39.3	137	154.2	69.8	197	221.8	101.0
18	20.3	10.0	78	87.8	39.8	138	155.4	70.3	198	222.9	101.5
19	21.4	10.5	79	88.9	40.3	139	156.5	70.8	199	224.0	102.0
20	22.5	11.0	80	90.1	40.8	140	157.6	71.3	200	225.2	102.6
21	23.6	11.5	81	91.2	41.3	141	158.7	71.8	201	226.3	103.1
22	24.8	12.0	82	92.3	41.8	142	159.9	72.3	202	227.4	103.7
23	25.9	12.5	83	93.4	42.3	143	161.0	72.9	203	228.5	104.2
24	27.0	13.0	84	94.6	42.8	144	162.1	73.4	204	229.7	104.7
25	28.1	13.5	85	95.7	43.4	145	163.2	73.9	205	230.8	105.3
26	29.3	14.0	86	96.8	43.9	146	164.4	74.4	206	231.9	105.8
27	30.4	14.5	87	97.9	44.4	147	165.5	74.9	207	233.0	106.3
28	31.5	15.0	88	99.1	44.9	148	166.6	75.5	208	234.2	106.8
29	32.7	15.5	89	100.2	45.4	149	167.7	76.0	209	235.3	107.4
30	33.8	16.0	90	101.3	45.9	150	168.9	76.5	210	236.4	107.9
31	34.9	16.5	91	102.4	46.4	151	170.0	77.0	211	237.6	108.4
32	36.0	17.0	92	103.6	46.9	152	171.1	77.5	212	238.7	109.0
33	37.2	17.5	93	104.7	47.4	153	172.3	78.1	213	239.8	109.5
34	38.3	18.0	94	105.8	47.9	154	173.4	78.6	214	240.9	110.0
35	39.4	18.5	95	107.0	48.4	155	174.5	79.1	215	242.1	110.6
36	40.5	18.9	96	108.1	48.9	156	175.6	79.6	216	243.2	111.1
37	41.7	19.4	97	109.2	49.4	157	176.8	80.1	217	244.3	111.6
38	42.8	19.9	98	110.3	49.9	158	177.9	80.7	218	245.4	112.1
39	43.9	20.4	99	111.5	50.4	159	179.0	81.2	219	246.6	112.7
40	45.0	20.9	100	112.6	50.9	160	180.1	81.7	220	247.7	113.2
41	46.2	21.4	101	113.7	51.4	161	181.3	82.2	221	248.7	113.7
42	47.3	21.9	102	114.8	51.9	162	182.4	82.7	222	249.9	114.2
43	48.4	22.4	103	116.0	52.4	163	183.5	83.3	223	251.0	114.8
44	49.5	22.9	104	117.1	52.9	164	184.6	83.8	224	252.4	115.3
45	50.7	23.4	105	118.2	53.5	165	185.8	84.3	225	253.3	115.9
46	51.8	23.9	106	119.3	54.0	166	186.9	84.8	226	254.4	116.4
47	52.9	24.4	107	120.5	54.5	167	188.0	85.3	227	255.6	116.9
48	54.0	24.9	108	121.6	55.0	168	189.1	85.9	228	256.7	117.4
49	55.2	25.4	109	122.7	55.5	169	190.3	86.4	229	257.8	118.0
50	56.3	25.9	110	123.8	56.0	170	191.4	86.9	230	258.9	118.5
51	57.4	26.4	111	125.0	56.5	171	192.5	87.4	231	260.1	119.0
52	58.5	26.9	112	126.1	57.0	172	193.6	87.9	232	261.2	119.6
53	59.7	27.4	113	127.2	57.5	173	194.8	88.5	233	262.3	120.1
54	60.8	27.9	114	128.3	58.0	174	195.9	89.0	234	263.4	120.7
55	61.9	28.4	115	129.6	58.6	175	197.0	89.5	235	264.6	121.2
56	63.0	28.8	116	130.6	59.1	176	198.1	90.0	236	265.7	121.7
57	64.2	29.3	117	131.7	59.6	177	199.3	90.5	237	266.8	122.2
58	65.3	29.8	118	132.8	60.1	178	200.4	91.1	238	268.0	122.8
59	66.4	30.3	119	134.0	60.6	179	201.5	91.6	239	269.1	123.4
60	67.6	30.8	120	135.1	61.1	180	202.6	92.1	240	270.2	123.9
61	68.7	31.3	121	136.2	61.6	181	203.8	92.6	241	271.3	124.4
62	69.8	31.8	122	137.4	62.1	182	204.9	93.1	242	272.5	125.0
63	70.9	32.3	123	138.5	62.6	183	206.0	93.7	243	273.6	125.5
64	72.1	32.8	124	139.6	63.1	184	207.1	94.2	244	274.7	126.0
65	73.2	33.3	125	140.7	63.7	185	208.3	94.7	245	275.8	126.6
66	74.3	33.8	126	141.9	64.2	186	209.4	95.2	246	277.0	127.1
67	75.4	34.3	127	143.0	64.7	187	210.5	95.7	247	278.1	127.6
68	76.6	34.8	128	144.1	65.2	188	211.7	96.3	248	279.2	128.1
69	77.7	35.3	129	145.2	65.7	189	212.8	96.8	249	280.3	128.7
70	78.8	35.8	130	146.4	66.2	190	213.9	97.3	250	281.5	129.2

TABLE 7.—ALLIHN'S TABLE.—Continued.

[Expressed in milligrams.]

COPPER	CU- PROUS OXID	DEX- TROSE	COPPER	CU- PROUS OXID	DEX- TROSE	COPPER	CU- PROUS OXID	DEX- TROSE	COPPER	CU- PROUS OXID	DEX- TROSE
251	282.6	129.7	306	344.5	159.8	361	406.4	190.6	416	469.4	222.2
252	283.7	130.3	307	345.6	160.4	362	407.6	191.1	417	469.5	222.8
253	284.8	130.8	308	346.8	160.9	363	408.7	191.7	418	470.6	223.2
254	286.0	131.4	309	347.9	161.5	364	409.8	192.3	419	471.8	223.9
255	287.1	131.9	310	349.0	162.0	365	410.9	192.9	420	472.9	224.5
256	288.2	132.4	311	350.1	162.6	366	412.1	193.4	421	474.0	225.1
257	289.3	133.0	312	351.3	163.1	367	413.2	194.0	422	475.6	225.7
258	290.5	133.5	313	352.4	163.7	368	414.3	194.6	423	476.2	226.3
259	291.6	134.1	314	353.5	164.2	369	415.4	195.1	424	477.4	226.9
260	292.7	134.6	315	354.6	164.8	370	416.6	195.7	425	478.5	227.5
261	293.8	135.1	316	355.8	165.3	371	417.7	196.3	426	479.6	228.0
262	295.0	135.7	317	356.9	165.9	372	418.8	196.8	427	480.7	228.6
263	296.1	136.2	318	358.0	166.4	373	420.0	197.4	428	481.9	229.2
264	297.2	136.8	319	359.1	167.0	374	421.1	198.0	429	483.0	229.8
265	298.3	137.3	320	360.3	167.5	375	422.2	198.6	430	484.1	230.4
266	299.5	137.8	321	361.4	168.1	376	423.3	199.1	431	485.3	231.0
267	300.6	138.4	322	362.5	168.6	377	424.5	199.7	432	486.4	231.6
268	301.7	138.9	323	363.7	169.2	378	425.6	200.3	433	487.5	232.2
269	302.8	139.5	324	364.8	169.7	379	426.7	200.8	434	488.6	232.8
270	304.0	140.0	325	365.9	170.3	380	427.8	201.4	435	489.7	233.4
271	305.1	140.6	326	367.0	170.9	381	429.0	202.0	436	490.9	233.9
272	306.2	141.1	327	368.2	171.4	382	430.1	202.5	437	492.0	234.5
273	307.3	141.7	328	369.3	172.0	383	431.2	203.1	438	493.1	235.1
274	308.5	142.2	329	370.4	172.5	384	432.3	203.7	439	494.3	235.7
275	309.6	142.8	330	371.5	173.1	385	433.5	204.3	440	495.4	236.3
276	310.7	143.3	331	372.7	173.7	386	434.6	204.8	441	496.5	236.9
277	311.9	143.9	332	373.8	174.2	387	435.7	205.4	442	497.6	237.5
278	313.0	144.4	333	374.9	174.8	388	436.8	206.0	443	498.8	238.1
279	314.1	145.0	334	376.0	175.3	389	438.0	206.5	444	499.9	238.7
280	315.2	145.5	335	377.2	175.9	390	439.1	207.1	445	501.0	239.3
281	316.4	146.1	336	378.3	176.5	391	440.2	207.7	446	502.1	239.8
282	317.5	146.6	337	379.4	177.0	392	441.3	208.3	447	503.2	240.4
283	318.6	147.2	338	380.5	177.6	393	442.4	208.8	448	504.4	241.0
284	319.7	147.7	339	381.7	178.1	394	443.6	209.4	449	505.5	241.6
285	320.9	148.3	340	382.8	178.7	395	444.7	210.0	450	506.6	242.2
286	322.0	148.8	341	383.9	179.3	396	445.9	210.6	451	507.8	242.8
287	323.1	149.4	342	385.0	179.8	397	447.0	211.2	452	508.9	243.4
288	324.2	149.9	343	386.2	180.4	398	448.1	211.7	453	510.0	244.0
289	325.4	150.5	344	387.3	180.9	399	449.2	212.3	454	511.1	244.6
290	326.5	151.0	345	388.4	181.5	400	450.3	212.9	455	512.3	245.2
291	327.4	151.6	346	389.6	182.1	401	451.5	213.5	456	513.4	245.7
292	328.7	152.1	347	390.7	182.6	402	452.6	214.1	457	514.5	246.3
293	329.9	152.7	348	391.8	183.2	403	453.7	214.6	458	515.6	246.9
294	331.0	153.2	349	392.9	183.7	404	454.8	215.2	459	516.8	247.5
295	332.1	153.8	350	394.0	184.3	405	456.0	215.8	460	517.9	248.1
296	333.3	154.3	351	395.2	184.9	406	457.1	216.4	461	519.0	248.7
297	334.4	154.9	352	396.3	185.4	407	458.2	217.0	462	520.1	249.3
298	335.5	155.4	353	397.4	186.0	408	459.4	217.5	463	521.3	249.9
299	336.6	156.0	354	398.6	186.6	409	460.5	218.1			
300	337.8	156.5	355	399.7	187.2	410	461.6	218.7			
301	338.9	157.1	356	400.8	187.7	411	462.7	219.3			
302	340.0	157.6	357	401.9	188.3	412	463.8	219.9			
303	341.1	158.2	358	403.1	188.9	413	465.0	220.4			
304	342.3	158.7	359	404.2	189.4	414	466.1	221.0			
305	343.4	159.3	360	405.3	190.0	415	467.2	221.6			

56

REDUCING SUGARS OTHER THAN DEXTROSE.

Proceed as directed under 54 and multiply the weight of dextrose found in 55 by the following factors:

Levulose,	1.093;
Invert sugar,	1.046;
Arabinose,	0.969;
Xylose,	1.017;
Galactose,	1.114.

TOTAL SUGARS.¹¹

(Applicable to cattle foods.)

57

PREPARATION OF SOLUTION.

Place 12 grams of the material in a 300 cc. graduated flask, if the substance has an acid reaction add 1-3 grams of calcium carbonate, and boil on a steam bath for 1 hour with 150 cc. of 50% alcohol by volume, using a small funnel in the neck of the flask to condense the vapor. Cool, and allow the mixture to stand several hours, preferably overnight. Make up to volume with neutral 95% alcohol, mix thoroughly, allow to settle, transfer 200 cc. to a beaker with a pipette, and evaporate on a steam bath to a volume of 20-30 cc.

Do not evaporate to dryness, a little alcohol in the residue doing no harm. Transfer to a 100 cc. graduated flask, and rinse the beaker thoroughly with water, adding the rinsings to the contents of the flask. Add enough saturated neutral lead acetate solution to produce a flocculent precipitate, shake thoroughly and allow to stand 15 minutes. Make up to the mark with water, mix thoroughly, and filter through a dry filter. Add sufficient anhydrous sodium carbonate to the filtrate to precipitate all the lead, again filter through a dry paper and test the filtrate with a little anhydrous sodium carbonate to make sure that all the lead has been removed.

58

DETERMINATION OF REDUCING SUGARS.

Proceed as directed under 26 or 29-34 respectively, employing the Soxhlet modification of Fehling's solution and using 25 cc. of the solution (representing 2 grams of the sample), prepared as directed in 57. Express the results as dextrose or invert sugar.

59

SUCROSE.

Introduce 50 cc. of the solution, prepared as directed in 57, into a 100 cc. graduated flask, add a piece of litmus paper, neutralize with acetic acid, add 5 cc. of concentrated hydrochloric acid and allow the inversion to proceed at room temperature as directed under 14 or 16. When inversion is complete, transfer the solution to a beaker, neutralize with sodium carbonate, return the solution to the 100 cc. flask, dilute to the mark with water, filter if necessary and determine reducing sugars in 50 cc. of the solution (representing 2 grams of the sample) as directed in 58, and calculate the results as invert sugar. Subtract the per cent of reducing sugars before inversion from the per cent of total sugar after inversion, both calculated as invert sugar, and multiply the difference by 0.95 to obtain the per cent of sucrose present.

Since the insoluble material of grain or cattle food occupies some space in the flask as originally made up, it is necessary to correct for this volume. Results of a large number of determinations on various materials have shown the average volume of 12 grams of material to be 9 cc., and therefore to obtain the true amount of sugars present all results must be multiplied by the factor 0.97.

STARCH.

60 *Direct Acid Hydrolysis (Modified Sachsse Method).—Official.*

(In this method there will be included as starch the pentosans and other carbohydrate bodies present which undergo hydrolysis and conversion into reducing sugars on boiling with hydrochloric acid.)

Stir a quantity of the sample, representing 2.5–3 grams of the dry material, in a beaker with 50 cc. of cold water for an hour. Transfer to a filter and wash with 250 cc. of cold water. Heat the insoluble residue for 2½ hours with 200 cc. of water and 20 cc. of hydrochloric acid (sp. gr. 1.125) in a flask provided with a reflux condenser. Cool, and nearly neutralize with sodium hydroxid. Complete the volume to 250 cc., filter, and determine the dextrose in an aliquot of the filtrate as directed under 52 or 54. The weight of the dextrose obtained multiplied by 0.90 gives the weight of starch.

The factor 0.9 is the theoretical ratio between starch and glucose but, according to Noyes¹² and other investigators, the factor 0.93 more nearly approaches the actual yield.

*Diastase Method with Subsequent Acid Hydrolysis.—Tentative.***61**

REAGENT.

Malt extract.—Digest 10 grams of fresh, finely ground malt for 2–3 hours at ordinary temperature with 200 cc. of water and filter. Determine the amount of dextrose in a given quantity of the filtrate after boiling with acid, etc., as in the starch determination, and make the proper correction in the subsequent determination.

62

DETERMINATION.

Extract a convenient quantity of the substance (ground to an impalpable powder and representing 4–5 grams of the dry material) on a hardened filter with 5 successive portions of 10 cc. of ether; wash with 150 cc. of 10% alcohol and then with a little strong alcohol. Place the residue in a beaker with 50 cc. of water, immerse the beaker in boiling water, and stir constantly for 15 minutes or until all the starch is gelatinized; cool to 55°C., add 20 cc. of malt extract, and maintain at this temperature for an hour. Heat again to boiling for a few minutes, cool to 55°C., add 20 cc. of malt extract, and maintain at this temperature for an hour or until the residue treated with iodine shows no blue color upon microscopic examination. Cool, make up directly to 250 cc., and filter. Place 200 cc. of the filtrate in a flask with 20 cc. of hydrochloric acid (sp. gr. 1.125); connect with a reflux condenser and heat in a boiling water bath for 2½ hours. Cool, nearly neutralize with sodium hydroxid solution, finish the neutralization with sodium carbonate solution, and make up to 500 cc. Mix the solution well, pour through a dry filter, and determine the dextrose in an aliquot as directed under 52 or 54. Conduct a blank determination upon the same volume of the malt extract as used upon the sample and correct the weight of reduced copper accordingly. The weight of the dextrose obtained multiplied by 0.90 gives the weight of starch.

PENTOSANS.—TENTATIVE.

63

REAGENT.

Phloroglucin.—Dissolve a small quantity of the phloroglucin in a few drops of acetic anhydrid, heat almost to boiling, and add a few drops of concentrated sul-

phuric acid. A violet color indicates the presence of diresorcin. A phloroglucin which gives more than a faint coloration may be purified by the following method:

Heat in a beaker about 300 cc. of hydrochloric acid (sp. gr. 1.06) and 11 grams of commercial phloroglucin, added in small quantities at a time, stirring constantly until it has almost entirely dissolved. Pour the hot solution into a sufficient quantity of the same hydrochloric acid (cold) to make the volume 1500 cc. Allow it to stand at least overnight, preferably several days, to permit the diresorcin to crystallize out. Filter immediately before using. A yellow tint does not interfere with its usefulness. In using it, add the volume containing the required amount to the distillate.

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DETERMINATION.

Place a quantity of the material, 2-5 grams, chosen so that the weight of phloroglucin obtained shall not exceed 0.300 gram, in a 300 cc. distillation flask, together with 100 cc. of 12% hydrochloric acid (sp. gr. 1.06), and several pieces of recently heated pumice stone. Place the flask on a wire gauze, connect with a condenser, and heat, rather gently at first, and regulate so as to distil over 30 cc. in about 10 minutes, the distillate passing through a small filter paper. Replace the 30 cc. distilled by a like quantity of the dilute acid, added by means of a separatory funnel in such a manner as to wash down the particles adhering to the sides of the flask, and continue the process until the distillate amounts to 360 cc. To the total distillate add gradually a quantity of phloroglucin dissolved in 12% hydrochloric acid and stir thoroughly the resulting mixture. The amount of phloroglucin used should be about double that of the furfural expected. The solution turns first yellow, then green, and very soon an amorphous greenish precipitate appears, which grows darker rapidly, till it becomes finally almost black. Make the solution up to 400 cc. with 12% hydrochloric acid, and allow to stand overnight.

Filter the amorphous black precipitate into a tared Gooch crucible through an asbestos mat, wash carefully with 150 cc. of water in such a way that the water is not entirely removed from the crucible until the very last, then dry for 4 hours at the temperature of boiling water, cool and weigh in a weighing bottle, the increase in weight being reckoned as furfural phloroglucin. To calculate the furfural, pentose, or pentosan from the phloroglucin, use the following formulas given by Kröber:

(1) For a weight of phloroglucin, designated by "a" in the following formulas, under 0.03 gram,

$$\text{Furfural} = (a + 0.0052) \times 0.5170.$$

$$\text{Pentoses} = (a + 0.0052) \times 1.0170.$$

$$\text{Pentosans} = (a + 0.0052) \times 0.8949.$$

In the above and also in the following formulas, the factor 0.0052 represents the weight of phloroglucin which remains dissolved in the 400 cc. of acid solution.

(2) For a weight of phloroglucin "a" over 0.300 gram,

$$\text{Furfural} = (a + 0.0052) \times 0.5180.$$

$$\text{Pentoses} = (a + 0.0052) \times 1.0026.$$

$$\text{Pentosans} = (a + 0.0052) \times 0.8824.$$

For a weight of phloroglucin "a" between 0.03 and 0.300 gram use Kröber's table, 65, or the following formulas in which the factors were calculated from Kröber's tables by C. A. Browne,¹³

$$\text{Furfural} = (a + 0.0052) \times 0.5185.$$

$$\text{Pentoses} = (a + 0.0052) \times 1.0075.$$

$$\text{Pentosans} = (a + 0.0052) \times 0.8866.$$

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TABLE 8.—KRÖBER'S TABLE.¹⁴*For Determining Pentoses and Pentosans.*

[Expressed in grams.]

FURFURAL FELOROGLUCE	FURFURAL	ARABINOSE	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.030	0.0182	0.0391	0.0344	0.0324	0.0285	0.0358	0.0315
0.031	0.0188	0.0402	0.0354	0.0333	0.0293	0.0368	0.0324
0.032	0.0193	0.0413	0.0363	0.0342	0.0301	0.0378	0.0333
0.033	0.0198	0.0424	0.0373	0.0352	0.0309	0.0388	0.0341
0.034	0.0203	0.0435	0.0383	0.0361	0.0317	0.0398	0.0350
0.035	0.0209	0.0446	0.0393	0.0370	0.0326	0.0408	0.0359
0.036	0.0214	0.0457	0.0402	0.0379	0.0334	0.0418	0.0368
0.037	0.0219	0.0468	0.0412	0.0388	0.0342	0.0428	0.0377
0.038	0.0224	0.0479	0.0422	0.0398	0.0350	0.0439	0.0386
0.039	0.0229	0.0490	0.0431	0.0407	0.0358	0.0449	0.0395
0.040	0.0235	0.0501	0.0441	0.0416	0.0366	0.0459	0.0404
0.041	0.0240	0.0512	0.0451	0.0425	0.0374	0.0469	0.0413
0.042	0.0245	0.0523	0.0460	0.0434	0.0382	0.0479	0.0422
0.043	0.0250	0.0534	0.0470	0.0443	0.0390	0.0489	0.0431
0.044	0.0255	0.0545	0.0480	0.0452	0.0398	0.0499	0.0440
0.045	0.0260	0.0556	0.0490	0.0462	0.0406	0.0509	0.0448
0.046	0.0266	0.0567	0.0499	0.0471	0.0414	0.0519	0.0457
0.047	0.0271	0.0578	0.0509	0.0480	0.0422	0.0529	0.0466
0.048	0.0276	0.0589	0.0519	0.0489	0.0430	0.0539	0.0475
0.049	0.0281	0.0600	0.0528	0.0498	0.0438	0.0549	0.0484
0.050	0.0286	0.0611	0.0538	0.0507	0.0446	0.0559	0.0492
0.051	0.0292	0.0622	0.0548	0.0516	0.0454	0.0569	0.0501
0.052	0.0297	0.0633	0.0557	0.0525	0.0462	0.0579	0.0510
0.053	0.0302	0.0644	0.0567	0.0534	0.0470	0.0589	0.0519
0.054	0.0307	0.0655	0.0576	0.0543	0.0478	0.0599	0.0528
0.055	0.0312	0.0666	0.0586	0.0553	0.0486	0.0610	0.0537
0.056	0.0318	0.0677	0.0596	0.0562	0.0494	0.0620	0.0546
0.057	0.0323	0.0688	0.0605	0.0571	0.0502	0.0630	0.0555
0.058	0.0328	0.0699	0.0615	0.0580	0.0510	0.0640	0.0564
0.059	0.0333	0.0710	0.0624	0.0589	0.0518	0.0650	0.0573
0.060	0.0338	0.0721	0.0634	0.0598	0.0526	0.0660	0.0581
0.061	0.0344	0.0732	0.0644	0.0607	0.0534	0.0670	0.0590
0.062	0.0349	0.0743	0.0653	0.0616	0.0542	0.0680	0.0599
0.063	0.0354	0.0754	0.0663	0.0626	0.0550	0.0690	0.0608
0.064	0.0359	0.0765	0.0673	0.0635	0.0558	0.0700	0.0617
0.065	0.0364	0.0776	0.0683	0.0644	0.0567	0.0710	0.0625
0.066	0.0370	0.0787	0.0692	0.0653	0.0575	0.0720	0.0634
0.067	0.0375	0.0798	0.0702	0.0662	0.0583	0.0730	0.0643
0.068	0.0380	0.0809	0.0712	0.0672	0.0591	0.0741	0.0652
0.069	0.0385	0.0820	0.0721	0.0681	0.0599	0.0751	0.0661
0.070	0.0390	0.0831	0.0731	0.0690	0.0607	0.0761	0.0670
0.071	0.0396	0.0842	0.0741	0.0699	0.0615	0.0771	0.0679
0.072	0.0401	0.0853	0.0750	0.0708	0.0623	0.0781	0.0688
0.073	0.0406	0.0864	0.0760	0.0717	0.0631	0.0791	0.0697
0.074	0.0411	0.0875	0.0770	0.0726	0.0639	0.0801	0.0706

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TABLE 8.—KRÖBER'S TABLE.—Continued.

[Expressed in grams.]

FURFURAL PELOROGUCID	FURFURAL	ARABINOSE	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.075	0.0416	0.0886	0.0780	0.0736	0.0647	0.0811	0.0714
0.076	0.0422	0.0897	0.0789	0.0745	0.0655	0.0821	0.0722
0.077	0.0427	0.0908	0.0799	0.0754	0.0663	0.0831	0.0731
0.078	0.0432	0.0919	0.0809	0.0763	0.0671	0.0841	0.0740
0.079	0.0437	0.0930	0.0818	0.0772	0.0679	0.0851	0.0749
0.080	0.0442	0.0941	0.0828	0.0781	0.0687	0.0861	0.0758
0.081	0.0448	0.0952	0.0838	0.0790	0.0695	0.0871	0.0767
0.082	0.0453	0.0963	0.0847	0.0799	0.0703	0.0881	0.0776
0.083	0.0458	0.0974	0.0857	0.0808	0.0711	0.0891	0.0785
0.084	0.0463	0.0985	0.0867	0.0817	0.0719	0.0901	0.0794
0.085	0.0468	0.0996	0.0877	0.0827	0.0727	0.0912	0.0803
0.086	0.0474	0.1007	0.0886	0.0836	0.0735	0.0922	0.0812
0.087	0.0479	0.1018	0.0896	0.0845	0.0743	0.0932	0.0821
0.088	0.0484	0.1029	0.0906	0.0854	0.0751	0.0942	0.0830
0.089	0.0489	0.1040	0.0915	0.0863	0.0759	0.0952	0.0838
0.090	0.0494	0.1051	0.0925	0.0872	0.0767	0.0962	0.0847
0.091	0.0499	0.1062	0.0935	0.0881	0.0775	0.0972	0.0856
0.092	0.0505	0.1073	0.0944	0.0890	0.0783	0.0982	0.0865
0.093	0.0510	0.1084	0.0954	0.0900	0.0791	0.0992	0.0874
0.094	0.0515	0.1095	0.0964	0.0909	0.0800	0.1002	0.0883
0.095	0.0520	0.1106	0.0974	0.0918	0.0808	0.1012	0.0891
0.096	0.0525	0.1117	0.0983	0.0927	0.0816	0.1022	0.0899
0.097	0.0531	0.1128	0.0993	0.0936	0.0824	0.1032	0.0908
0.098	0.0536	0.1139	0.1003	0.0946	0.0832	0.1043	0.0917
0.099	0.0541	0.1150	0.1012	0.0955	0.0840	0.1053	0.0926
0.100	0.0546	0.1161	0.1022	0.0964	0.0848	0.1063	0.0935
0.101	0.0551	0.1171	0.1032	0.0973	0.0856	0.1073	0.0944
0.102	0.0557	0.1182	0.1041	0.0982	0.0864	0.1083	0.0953
0.103	0.0562	0.1193	0.1051	0.0991	0.0872	0.1093	0.0962
0.104	0.0567	0.1204	0.1060	0.1000	0.0880	0.1103	0.0971
0.105	0.0572	0.1215	0.1070	0.1010	0.0888	0.1113	0.0979
0.106	0.0577	0.1226	0.1080	0.1019	0.0896	0.1123	0.0988
0.107	0.0582	0.1237	0.1089	0.1028	0.0904	0.1133	0.0997
0.108	0.0588	0.1248	0.1099	0.1037	0.0912	0.1143	0.1006
0.109	0.0593	0.1259	0.1108	0.1046	0.0920	0.1153	0.1015
0.110	0.0598	0.1270	0.1118	0.1055	0.0928	0.1163	0.1023
0.111	0.0603	0.1281	0.1128	0.1064	0.0936	0.1173	0.1032
0.112	0.0608	0.1292	0.1137	0.1073	0.0944	0.1183	0.1041
0.113	0.0614	0.1303	0.1147	0.1082	0.0952	0.1193	0.1050
0.114	0.0619	0.1314	0.1156	0.1091	0.0960	0.1203	0.1059
0.115	0.0624	0.1325	0.1166	0.1101	0.0968	0.1213	0.1067
0.116	0.0629	0.1336	0.1176	0.1110	0.0976	0.1223	0.1076
0.117	0.0634	0.1347	0.1185	0.1119	0.0984	0.1233	0.1085
0.118	0.0640	0.1358	0.1195	0.1128	0.0992	0.1243	0.1094
0.119	0.0645	0.1369	0.1204	0.1137	0.1000	0.1253	0.1103

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TABLE 8.—KRÖBER'S TABLE.—Continued.

[Expressed in grams.]

FURFURAL PHENOLGLUCID	FURFURAL	ARABINOS	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.120	0.0650	0.1380	0.1214	0.1146	0.1008	0.1263	0.1111
0.121	0.0655	0.1391	0.1224	0.1155	0.1016	0.1273	0.1120
0.122	0.0660	0.1402	0.1233	0.1164	0.1024	0.1283	0.1129
0.123	0.0665	0.1413	0.1243	0.1173	0.1032	0.1293	0.1138
0.124	0.0671	0.1424	0.1253	0.1182	0.1040	0.1303	0.1147
0.125	0.0676	0.1435	0.1263	0.1192	0.1049	0.1314	0.1156
0.126	0.0681	0.1446	0.1272	0.1201	0.1057	0.1324	0.1165
0.127	0.0686	0.1457	0.1282	0.1210	0.1065	0.1334	0.1174
0.128	0.0691	0.1468	0.1292	0.1219	0.1073	0.1344	0.1183
0.129	0.0697	0.1479	0.1301	0.1228	0.1081	0.1354	0.1192
0.130	0.0702	0.1490	0.1311	0.1237	0.1089	0.1364	0.1201
0.131	0.0707	0.1501	0.1321	0.1246	0.1097	0.1374	0.1210
0.132	0.0712	0.1512	0.1330	0.1255	0.1105	0.1384	0.1219
0.133	0.0717	0.1523	0.1340	0.1264	0.1113	0.1394	0.1227
0.134	0.0723	0.1534	0.1350	0.1273	0.1121	0.1404	0.1236
0.135	0.0728	0.1545	0.1360	0.1283	0.1129	0.1414	0.1244
0.136	0.0733	0.1556	0.1369	0.1292	0.1137	0.1424	0.1253
0.137	0.0738	0.1567	0.1379	0.1301	0.1145	0.1434	0.1262
0.138	0.0743	0.1578	0.1389	0.1310	0.1153	0.1444	0.1271
0.139	0.0748	0.1589	0.1398	0.1319	0.1161	0.1454	0.1280
0.140	0.0754	0.1600	0.1408	0.1328	0.1169	0.1464	0.1288
0.141	0.0759	0.1611	0.1418	0.1337	0.1177	0.1474	0.1297
0.142	0.0764	0.1622	0.1427	0.1346	0.1185	0.1484	0.1306
0.143	0.0769	0.1633	0.1437	0.1355	0.1193	0.1494	0.1315
0.144	0.0774	0.1644	0.1447	0.1364	0.1201	0.1504	0.1324
0.145	0.0780	0.1655	0.1457	0.1374	0.1209	0.1515	0.1333
0.146	0.0785	0.1666	0.1466	0.1383	0.1217	0.1525	0.1342
0.147	0.0790	0.1677	0.1476	0.1392	0.1225	0.1535	0.1351
0.148	0.0795	0.1688	0.1486	0.1401	0.1233	0.1545	0.1360
0.149	0.0800	0.1699	0.1495	0.1410	0.1241	0.1555	0.1369
0.150	0.0805	0.1710	0.1505	0.1419	0.1249	0.1565	0.1377
0.151	0.0811	0.1721	0.1515	0.1428	0.1257	0.1575	0.1386
0.152	0.0816	0.1732	0.1524	0.1437	0.1265	0.1585	0.1395
0.153	0.0821	0.1743	0.1534	0.1446	0.1273	0.1595	0.1404
0.154	0.0826	0.1754	0.1544	0.1455	0.1281	0.1605	0.1413
0.155	0.0831	0.1765	0.1554	0.1465	0.1289	0.1615	0.1421
0.156	0.0837	0.1776	0.1563	0.1474	0.1297	0.1625	0.1430
0.157	0.0842	0.1787	0.1573	0.1483	0.1305	0.1635	0.1439
0.158	0.0847	0.1798	0.1583	0.1492	0.1313	0.1645	0.1448
0.159	0.0852	0.1809	0.1592	0.1501	0.1321	0.1655	0.1457
0.160	0.0857	0.1820	0.1602	0.1510	0.1329	0.1665	0.1465
0.161	0.0863	0.1831	0.1612	0.1519	0.1337	0.1675	0.1474
0.162	0.0868	0.1842	0.1621	0.1528	0.1345	0.1685	0.1483
0.163	0.0873	0.1853	0.1631	0.1537	0.1353	0.1695	0.1492
0.164	0.0878	0.1864	0.1640	0.1546	0.1361	0.1705	0.1501

TABLE 8.—KRÖBER'S TABLE.—Continued.

[Expressed in grams.]

FURFURAL PHENOLGLUCID	FURFURAL	ARABINOSE	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.165	0.0883	0.1875	0.1650	0.1556	0.1369	0.1716	0.1510
0.166	0.0888	0.1886	0.1660	0.1565	0.1377	0.1726	0.1519
0.167	0.0894	0.1897	0.1669	0.1574	0.1385	0.1736	0.1528
0.168	0.0899	0.1908	0.1679	0.1583	0.1393	0.1746	0.1537
0.169	0.0904	0.1919	0.1688	0.1592	0.1401	0.1756	0.1546
0.170	~0.0909	0.1930	0.1698	0.1601	0.1409	0.1766	0.1554
0.171	0.0914	0.1941	0.1708	0.1610	0.1417	0.1776	0.1563
0.172	0.0920	0.1952	0.1717	0.1619	0.1425	0.1786	0.1572
0.173	0.0925	0.1963	0.1727	0.1628	0.1433	0.1796	0.1581
0.174	0.0930	0.1974	0.1736	0.1637	0.1441	0.1806	0.1590
0.175	0.0935	0.1985	0.1746	0.1647	0.1449	0.1816	0.1598
0.176	0.0940	0.1996	0.1756	0.1656	0.1457	0.1826	0.1607
0.177	0.0946	0.2007	0.1765	0.1665	0.1465	0.1836	0.1616
0.178	0.0951	0.2018	0.1775	0.1674	0.1473	0.1846	0.1625
0.179	0.0956	0.2029	0.1784	0.1683	0.1481	0.1856	0.1634
0.180	0.0961	0.2039	0.1794	0.1692	0.1489	0.1866	0.1642
0.181	0.0966	0.2050	0.1804	0.1701	0.1497	0.1876	0.1651
0.182	0.0971	0.2061	0.1813	0.1710	0.1505	0.1886	0.1660
0.183	0.0977	0.2072	0.1823	0.1719	0.1513	0.1896	0.1669
0.184	0.0982	0.2082	0.1832	0.1728	0.1521	0.1906	0.1678
0.185	0.0987	0.2093	0.1842	0.1738	0.1529	0.1916	0.1686
0.186	0.0992	0.2104	0.1851	0.1747	0.1537	0.1926	0.1695
0.187	0.0997	0.2115	0.1861	0.1756	0.1545	0.1936	0.1704
0.188	0.1003	0.2126	0.1870	0.1765	0.1553	0.1946	0.1712
0.189	0.1008	0.2136	0.1880	0.1774	0.1561	0.1955	0.1721
0.190	0.1013	0.2147	0.1889	0.1783	0.1569	0.1965	0.1729
0.191	0.1018	0.2158	0.1899	0.1792	0.1577	0.1975	0.1738
0.192	0.1023	0.2168	0.1908	0.1801	0.1585	0.1985	0.1747
0.193	0.1028	0.2179	0.1918	0.1810	0.1593	0.1995	0.1756
0.194	0.1034	0.2190	0.1927	0.1819	0.1601	0.2005	0.1764
0.195	0.1039	0.2201	0.1937	0.1829	0.1609	0.2015	0.1773
0.196	0.1044	0.2212	0.1946	0.1838	0.1617	0.2025	0.1782
0.197	0.1049	0.2222	0.1956	0.1847	0.1625	0.2035	0.1791
0.198	0.1054	0.2233	0.1965	0.1856	0.1633	0.2045	0.1800
0.199	0.1059	0.2244	0.1975	0.1865	0.1641	0.2055	0.1808
0.200	0.1065	0.2255	0.1984	0.1874	0.1649	0.2065	0.1817
0.201	0.1070	0.2266	0.1994	0.1883	0.1657	0.2075	0.1826
0.202	0.1075	0.2276	0.2003	0.1892	0.1665	0.2085	0.1835
0.203	0.1080	0.2287	0.2013	0.1901	0.1673	0.2095	0.1844
0.204	0.1085	0.2298	0.2022	0.1910	0.1681	0.2105	0.1853
0.205	0.1090	0.2309	0.2032	0.1920	0.1689	0.2115	0.1861
0.206	0.1096	0.2320	0.2041	0.1929	0.1697	0.2125	0.1869
0.207	0.1101	0.2330	0.2051	0.1938	0.1705	0.2134	0.1878
0.208	0.1106	0.2341	0.2060	0.1947	0.1713	0.2144	0.1887
0.209	0.1111	0.2352	0.2069	0.1956	0.1721	0.2154	0.1896

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TABLE 8.—KRÖBER'S TABLE.—Continued.

[Expressed in grams.]

FURFURAL PELOROGLUCID	FURFURAL	ARABINOSE	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.210	0.1116	0.2363	0.2079	0.1965	0.1729	0.2164	0.1904
0.211	0.1121	0.2374	0.2089	0.1975	0.1737	0.2174	0.1913
0.212	0.1127	0.2384	0.2098	0.1984	0.1745	0.2184	0.1922
0.213	0.1132	0.2395	0.2108	0.1993	0.1753	0.2194	0.1931
0.214	0.1137	0.2406	0.2117	0.2002	0.1761	0.2204	0.1940
0.215	0.1142	0.2417	0.2127	0.2011	0.1770	0.2214	0.1948
0.216	0.1147	0.2428	0.2136	0.2020	0.1778	0.2224	0.1957
0.217	0.1152	0.2438	0.2146	0.2029	0.1786	0.2234	0.1966
0.218	0.1158	0.2449	0.2155	0.2038	0.1794	0.2244	0.1974
0.219	0.1163	0.2460	0.2165	0.2047	0.1802	0.2254	0.1983
0.220	0.1168	0.2471	0.2174	0.2057	0.1810	0.2264	0.1992
0.221	0.1173	0.2482	0.2184	0.2066	0.1818	0.2274	0.2001
0.222	0.1178	0.2492	0.2193	0.2075	0.1826	0.2284	0.2010
0.223	0.1183	0.2503	0.2203	0.2084	0.1834	0.2294	0.2019
0.224	0.1189	0.2514	0.2212	0.2093	0.1842	0.2304	0.2028
0.225	0.1194	0.2525	0.2222	0.2102	0.1850	0.2314	0.2037
0.226	0.1199	0.2536	0.2232	0.2111	0.1858	0.2324	0.2046
0.227	0.1204	0.2546	0.2241	0.2121	0.1866	0.2334	0.2054
0.228	0.1209	0.2557	0.2251	0.2130	0.1874	0.2344	0.2063
0.229	0.1214	0.2568	0.2260	0.2139	0.1882	0.2354	0.2072
0.230	0.1220	0.2579	0.2270	0.2148	0.1890	0.2364	0.2081
0.231	0.1225	0.2590	0.2280	0.2157	0.1898	0.2374	0.2089
0.232	0.1230	0.2600	0.2289	0.2166	0.1906	0.2383	0.2097
0.233	0.1235	0.2611	0.2299	0.2175	0.1914	0.2393	0.2106
0.234	0.1240	0.2622	0.2308	0.2184	0.1922	0.2403	0.2115
0.235	0.1245	0.2633	0.2318	0.2193	0.1930	0.2413	0.2124
0.236	0.1251	0.2644	0.2327	0.2202	0.1938	0.2423	0.2132
0.237	0.1256	0.2654	0.2337	0.2211	0.1946	0.2433	0.2141
0.238	0.1261	0.2665	0.2346	0.2220	0.1954	0.2443	0.2150
0.239	0.1266	0.2676	0.2356	0.2229	0.1962	0.2453	0.2159
0.240	0.1271	0.2687	0.2365	0.2239	0.1970	0.2463	0.2168
0.241	0.1276	0.2698	0.2375	0.2248	0.1978	0.2473	0.2176
0.242	0.1281	0.2708	0.2384	0.2257	0.1986	0.2483	0.2185
0.243	0.1287	0.2719	0.2394	0.2266	0.1994	0.2493	0.2194
0.244	0.1292	0.2730	0.2403	0.2275	0.2002	0.2503	0.2203
0.245	0.1297	0.2741	0.2413	0.2284	0.2010	0.2513	0.2212
0.246	0.1302	0.2752	0.2422	0.2293	0.2018	0.2523	0.2220
0.247	0.1307	0.2762	0.2432	0.2302	0.2026	0.2533	0.2229
0.248	0.1312	0.2773	0.2441	0.2311	0.2034	0.2543	0.2238
0.249	0.1318	0.2784	0.2451	0.2320	0.2042	0.2553	0.2247
0.250	0.1323	0.2795	0.2460	0.2330	0.2050	0.2563	0.2256
0.251	0.1328	0.2806	0.2470	0.2339	0.2058	0.2573	0.2264
0.252	0.1333	0.2816	0.2479	0.2348	0.2066	0.2582	0.2272
0.253	0.1338	0.2827	0.2489	0.2357	0.2074	0.2592	0.2281
0.254	0.1343	0.2838	0.2498	0.2366	0.2082	0.2602	0.2290

TABLE 8.—KRÖBER'S TABLE.—Continued.

[Expressed in grams.]

FURFURAL FELOROGUCID	FURFURAL	ARABINOSE	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.255	0.1349	0.2849	0.2508	0.2375	0.2090	0.2612	0.2299
0.256	0.1354	0.2860	0.2517	0.2384	0.2098	0.2622	0.2377
0.257	0.1359	0.2870	0.2526	0.2393	0.2106	0.2632	0.2316
0.258	0.1364	0.2881	0.2536	0.2402	0.2114	0.2642	0.2325
0.259	0.1369	0.2892	0.2545	0.2411	0.2122	0.2652	0.2334
0.260	0.1374	0.2903	0.2555	0.2420	0.2130	0.2662	0.2342
0.261	0.1380	0.2914	0.2565	0.2429	0.2138	0.2672	0.2351
0.262	0.1385	0.2924	0.2574	0.2438	0.2146	0.2681	0.2359
0.263	0.1390	0.2935	0.2584	0.2447	0.2154	0.2691	0.2368
0.264	0.1395	0.2946	0.2593	0.2456	0.2162	0.2701	0.2377
0.265	0.1400	0.2957	0.2603	0.2465	0.2170	0.2711	0.2385
0.266	0.1405	0.2968	0.2612	0.2474	0.2178	0.2721	0.2394
0.267	0.1411	0.2978	0.2622	0.2483	0.2186	0.2731	0.2403
0.268	0.1416	0.2989	0.2631	0.2492	0.2194	0.2741	0.2412
0.269	0.1421	0.3000	0.2641	0.2502	0.2202	0.2751	0.2421
0.270	0.1426	0.3011	0.2650	0.2511	0.2210	0.2761	0.2429
0.271	0.1431	0.3022	0.2660	0.2520	0.2218	0.2771	0.2438
0.272	0.1436	0.3032	0.2669	0.2529	0.2226	0.2781	0.2447
0.273	0.1442	0.3043	0.2679	0.2538	0.2234	0.2791	0.2456
0.274	0.1447	0.3054	0.2688	0.2547	0.2242	0.2801	0.2465
0.275	0.1452	0.3065	0.2698	0.2556	0.2250	0.2811	0.2473
0.276	0.1457	0.3076	0.2707	0.2565	0.2258	0.2821	0.2482
0.277	0.1462	0.3086	0.2717	0.2574	0.2266	0.2830	0.2490
0.278	0.1467	0.3097	0.2726	0.2583	0.2274	0.2840	0.2499
0.279	0.1473	0.3108	0.2736	0.2592	0.2282	0.2850	0.2508
0.280	0.1478	0.3119	0.2745	0.2602	0.2290	0.2861	0.2517
0.281	0.1483	0.3130	0.2755	0.2611	0.2298	0.2871	0.2526
0.282	0.1488	0.3140	0.2764	0.2620	0.2306	0.2880	0.2534
0.283	0.1493	0.3151	0.2774	0.2629	0.2314	0.2890	0.2543
0.284	0.1498	0.3162	0.2783	0.2638	0.2322	0.2900	0.2552
0.285	0.1504	0.3173	0.2793	0.2647	0.2330	0.2910	0.2561
0.286	0.1509	0.3184	0.2802	0.2656	0.2338	0.2920	0.2570
0.287	0.1514	0.3194	0.2812	0.2665	0.2346	0.2930	0.2578
0.288	0.1519	0.3205	0.2821	0.2674	0.2354	0.2940	0.2587
0.289	0.1524	0.3216	0.2831	0.2683	0.2362	0.2950	0.2596
0.290	0.1529	0.3227	0.2840	0.2693	0.2370	0.2960	0.2605
0.291	0.1535	0.3238	0.2850	0.2702	0.2378	0.2970	0.2614
0.292	0.1540	0.3248	0.2859	0.2711	0.2386	0.2980	0.2622
0.293	0.1545	0.3259	0.2868	0.2720	0.2394	0.2990	0.2631
0.294	0.1550	0.3270	0.2878	0.2729	0.2402	0.3000	0.2640
0.295	0.1555	0.3281	0.2887	0.2738	0.2410	0.3010	0.2649
0.296	0.1560	0.3292	0.2897	0.2747	0.2418	0.3020	0.2658
0.297	0.1566	0.3302	0.2906	0.2756	0.2426	0.3030	0.2666
0.298	0.1571	0.3313	0.2916	0.2765	0.2434	0.3040	0.2675
0.299	0.1576	0.3324	0.2925	0.2774	0.2442	0.3050	0.2684
0.300	0.1581	0.3335	0.2935	0.2784	0.2450	0.3060	0.2693

66

GALACTAN.—TENTATIVE.

Extract a convenient quantity of the substance, representing 2.5–3 grams of the dry material, on a hardened filter with 5 successive portions of 10 cc. of ether, place the extracted residue in a beaker, about 5.5 cm. in diameter and 7 cm. deep, together with 60 cc. of nitric acid of 1.15 sp. gr., and evaporate the solution to exactly one third its volume in a water bath at a temperature of 94°–96°C. After standing 24 hours, add 10 cc. of water to the precipitate, and allow it to stand another 24 hours. The mucic acid has in the meantime crystallized, but it is mixed with considerable material only partially oxidized by the nitric acid. Filter the solution through filter paper, wash with 30 cc. of water to remove as much of the nitric acid as possible, and replace the filter and contents in the beaker. Add 30 cc. of ammonium carbonate solution, consisting of 1 part ammonium carbonate, 19 parts water, and 1 part strong ammonium hydroxid, and heat the mixture on a water bath, at 80°C., for 15 minutes, with constant stirring. The ammonium carbonate takes up the mucic acid, forming soluble ammonium mucate. Wash the filter paper and contents several times with hot water by decantation, passing the washings through a filter paper, to which finally transfer the material and thoroughly wash. Evaporate the filtrate to dryness over a water bath, avoiding unnecessary heating which causes decomposition, add 5 cc. of nitric acid of 1.15 sp. gr., stir thoroughly the mixture and allow to stand for 30 minutes. The nitric acid decomposes the ammonium mucate, precipitating the mucic acid; collect this on a tared filter or Gooch, wash with 10–15 cc. of water, then with 60 cc. of alcohol, and a number of times with ether, dry at the temperature of boiling water for 3 hours, and weigh. Multiply the weight of the mucic acid by 1.33, which gives galactose, and multiply this product by 0.9 which gives galactan.

CRUDE FIBER.—OFFICIAL.

67

REAGENTS.

- (a) 1.25% sulphuric acid solution.—Exact strength, determined by titration.
- (b) 1.25% sodium hydroxid solution.—Exact strength, determined by titration.

68

DETERMINATION.

Extract a quantity of the substance, representing about 2 grams of the dry material, with ordinary ether, or use the residue from the determination of the ether extract. To this residue in a 500 cc. flask add 200 cc. of boiling 1.25% sulphuric acid; connect the flask with an inverted condenser, the tube of which passes only a short distance beyond the rubber stopper into the flask, or simply cover a tall conical flask, which is well suited for this determination, with a watch glass or short stemmed funnel, boil at once and continue boiling gently for 30 minutes. A blast of air conducted into the flask will serve to reduce the frothing of the liquid. Filter through linen and wash with boiling water until the washings are no longer acid; rinse the substance back into the flask with 200 cc. of boiling, 1.25% solution of sodium hydroxid, free or nearly free from sodium carbonate boil at once, and continue boiling gently for 30 minutes as directed above for the treatment with acid, filter at once rapidly, and wash with boiling water until the washings are neutral. The last filtration may be performed upon a Gooch crucible, a linen filter, or a tared filter paper. If a linen filter is used, rinse the crude fiber, after washing is completed, into a flat-bottomed platinum dish by means of a jet of water; evaporate to dryness on a steam bath, dry to constant weight at 110°C., weigh, incinerate completely, and weigh again. The loss in weight is considered to be crude fiber. If a tared filter

paper is used, weigh in a weighing bottle. In any case the crude fiber after drying to constant weight at 110°C. must be incinerated and the amount of the ash deducted from the original weight.

69

WATER-SOLUBLE ACIDITY OF FEEDS.—TENTATIVE.

Weigh 10 grams of the sample into a shaking bottle, add 200 cc. of water, and shake for 15 minutes. Filter the extract through a folded filter and take a 20 cc. aliquot (equivalent to 1 gram of sample) for the titration. Dilute with 50 cc. of water and titrate with N/10 sodium hydroxid, using phenolphthalein as indicator.

In reporting the acidity of feeds, state the results in terms of cc. of N/10 sodium hydroxid required for neutralization.

BIBLIOGRAPHY.

- ¹ Z. Ver. Zucker-Ind., 1900, **37** (I): 357; 1913, **63** (I): 25; J. Ind. Eng. Chem., 1913, **5**: 167.
- ² J. Am. Chem. Soc., 1914, **36**: 1566.
- ³ Ibid., 1906, **28**: 663; 1907, **29**: 541.
- ⁴ Ibid., 1902, **24**: 1082.
- ⁵ Z. Rubenzucker-Ind., 1885, **35** (N. F. **22**): 1012.
- ⁶ Ibid., 1889, **39** (N. F. **26**): 734.
- ⁷ Ibid., 1879, **29** (N. F. **16**): 1034.
- ⁸ Wein. Tables for the Quantitative Estimation of the Sugars. Translated by Frew. 1896, p. 26.
- ⁹ Ibid., p. 33.
- ¹⁰ Z. Rubenzucker-Ind., 1882, **32** (N. F. **19**): 606, 865.
- ¹¹ U. S. Bur. Chem. Circ. 71.
- ¹² J. Am. Chem. Soc., 1904, **26**: 266.
- ¹³ U. S. Bur. Chem. Bull. 73, p. 173.
- ¹⁴ J. Landw., 1900, **43**: 379.

IX. SACCHARINE PRODUCTS.

1

PREPARATION OF SAMPLE.—TENTATIVE.

(a) *Liquids (molasses, sirups, etc.)*.—Mix materials of this class thoroughly. If crystals of sugar are present, dissolve them either by heating gently or by weighing the whole mass, then adding water, heating until completely dissolved and after cooling, re-weighing. Calculate all results to the weight of the original substance.

(b) *Semisolids (jellies, jams, etc.)*.—Weigh 50 grams of the sample into a 250 cc. graduated flask. Treat with water, fill to the mark and mix thoroughly. If insoluble material remains, mix uniformly by shaking before taking aliquots for the various determinations.

(c) *Solids (sugar, confectionery, etc.)*.—Grind and mix thoroughly materials of this class to secure uniform samples.

MOISTURE.

DRYING METHODS.

2

SUGARS.—OFFICIAL.

Dry 2-5 grams in a flat dish (nickel, platinum, or aluminium) at the temperature of boiling water for 10 hours; cool in a desiccator and weigh; then dry again for an hour or until there is only a slight change in weight.

With some sugars, more especially those of large grain, there is danger of occlusion and retention of water. The International Commission for Unifying Methods of Sugar Analysis prescribe drying at 105°-110°C. for normal beet sugars. This temperature is sufficient to expel the last traces of occluded water and is not attended with sufficient decomposition to affect the weight of the product. The drying temperature should never exceed 110°C.

MASSECUITES, MOLASSES, AND OTHER LIQUID AND SEMILIQUID PRODUCTS.

3

Drying upon Pumice Stone.—Tentative.

Prepare pumice stone of two grades of fineness, one of which will pass through a 1 mm. sieve, the other through a 6 mm. sieve. Make the determination in flat metallic dishes or in shallow, flat-bottomed, weighing bottles. Place a layer of the fine pumice stone, 3 mm. in thickness, on the bottom of the dish, then a layer of the coarse pumice stone 6-10 mm. in thickness, dry and weigh. Dilute the sample with a weighed portion of water so that the diluted material shall contain 20-30% of solid matter. Weigh into the dish, prepared as described above, an amount of the diluted sample to yield, approximately, 1 gram of dry matter. If this weighing can not be made rapidly, use a weighing bottle provided with a cork through which a pipette passes. Dry in vacuo at 70°C. to constant weight, making trial weighings at intervals of 2 hours. For substances containing little or no levulose or other readily decomposable substance, the drying may be made in a water oven at the temperature of boiling water.

4

Drying upon Quartz Sand.—Tentative.

Digest pure quartz sand with strong hydrochloric acid, wash, dry, and ignite. Preserve in a stoppered bottle.

Place 6-7 grams of the prepared sand and a short stirring rod in a flat-bottomed dish. Dry thoroughly, cool in a desiccator, and weigh. Then add 3-4 grams of the molasses, mix with the sand (if necessary to thoroughly incorporate the two, add a little water), dry in a water oven at the temperature of boiling water for 8-10 hours, stirring at intervals of an hour, cool in a desiccator, and weigh. Stir, heat again for an hour, cool, and weigh. Repeat the heating and weighing until the loss of water in an hour is not greater than 3 mg.

AREOMETRIC METHODS.

(Not applicable to low-grade sugar products, molasses and other materials containing large amounts of non-sugar solids.)

SPECIFIC GRAVITY, WATER AND TOTAL SOLIDS.

5

By Means of a Spindle.—Official.

The density of juices, sirups, etc., is most conveniently determined by means of the Brix hydrometer. For rough work, or where less accuracy is desired, the Baumé hydrometer may be used. The Brix spindle should be graduated to tenths. The range of degrees recorded by each individual spindle should be as limited as possible. The solution should be as nearly as practicable of the same temperature as the air at the time of reading, and, if the variation from the temperature of the graduation of the spindle amounts to more than 1°, a correction must be applied according to the table under 6. Before taking the density of a juice, allow it to stand in the cylinder until all air bubbles have escaped, and until all fatty or waxy matter has come to the surface and been skimmed off. The cylinder should be large enough in diameter to allow the hydrometer to come to rest without touching the sides. A table of specific gravities at $\frac{20^{\circ}\text{C.}}{4^{\circ}}$ and per cent by weight of sucrose is given under 9, and a table for the comparison of specific gravities at $\frac{17.5^{\circ}\text{C.}}{17.5^{\circ}}$, degrees Brix (per cent by weight of sucrose), and degree Baumé is given under 8.

If the sample is too dense to determine the density directly, dilute a weighed portion with a weighed quantity of water, or dissolve a weighed portion and dilute to a known volume with water.

In the first instance the per cent of total solids is calculated by the following formula:

$$\text{Per cent of solids in the undiluted material} = \frac{WS}{W} \text{ in which}$$

S = per cent of solids in the diluted material;

W = weight of the diluted material;

w = weight of the sample taken for dilution.

When the dilution is made to a definite volume, the following formula is to be used:

$$\text{Per cent of solids in the undiluted material} = \frac{VDS}{W} \text{ in which}$$

V = volume of the diluted solution at a given temperature;

D = specific gravity of the diluted solution at the same temperature;

S = per cent of solids in the diluted solution at the same temperature;

W = weight of the sample taken for dilution at the same temperature.

If the spindle reading be made at any other temperature than 17.5°C., the result should be corrected according to the following:

6

TABLE 9.

For correction of the readings of the Briz spindle when made at other than the standard temperature, 17.5°C.

(For temperatures below 17.5°C. the correction is to be subtracted.)

TEMPERATURE	DEGREE BRIZ OF THE SOLUTION												
	0	5	10	15	20	25	30	35	40	50	60	70	75
°C.													
0	0.17	0.30	0.41	0.52	0.62	0.72	0.82	0.92	0.98	1.11	1.22	1.25	1.29
5	0.23	0.30	0.37	0.44	0.52	0.59	0.65	0.72	0.75	0.80	0.88	0.91	0.94
10	0.20	0.26	0.29	0.33	0.36	0.39	0.42	0.45	0.48	0.50	0.54	0.58	0.61
11	0.18	0.23	0.26	0.28	0.31	0.34	0.36	0.39	0.41	0.43	0.47	0.50	0.53
12	0.16	0.20	0.22	0.24	0.26	0.29	0.31	0.33	0.34	0.36	0.40	0.42	0.46
13	0.14	0.18	0.19	0.21	0.22	0.24	0.26	0.27	0.28	0.29	0.33	0.35	0.39
14	0.12	0.15	0.16	0.17	0.18	0.19	0.21	0.22	0.22	0.23	0.26	0.28	0.32
15	0.09	0.11	0.12	0.14	0.14	0.15	0.16	0.17	0.16	0.17	0.19	0.21	0.25
16	0.06	0.07	0.08	0.09	0.10	0.10	0.11	0.12	0.12	0.12	0.14	0.16	0.18
17	0.02	0.02	0.03	0.03	0.03	0.04	0.04	0.04	0.04	0.04	0.05	0.06	0.06
18	0.02	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.02
19	0.06	0.08	0.08	0.09	0.09	0.10	0.10	0.10	0.10	0.10	0.10	0.08	0.06
20	0.11	0.14	0.15	0.17	0.17	0.18	0.18	0.18	0.19	0.19	0.18	0.15	0.11
21	0.16	0.20	0.22	0.24	0.24	0.25	0.25	0.25	0.26	0.26	0.25	0.22	0.18
22	0.21	0.26	0.29	0.31	0.31	0.32	0.32	0.32	0.33	0.34	0.32	0.29	0.25
23	0.27	0.32	0.35	0.37	0.38	0.39	0.39	0.39	0.40	0.42	0.39	0.36	0.33
24	0.32	0.38	0.41	0.43	0.44	0.46	0.46	0.47	0.47	0.50	0.46	0.43	0.40
25	0.37	0.44	0.47	0.49	0.51	0.53	0.54	0.55	0.55	0.58	0.54	0.51	0.48
26	0.43	0.50	0.54	0.56	0.58	0.60	0.61	0.62	0.62	0.66	0.62	0.58	0.55
27	0.49	0.57	0.61	0.63	0.65	0.68	0.68	0.69	0.70	0.74	0.70	0.65	0.62
28	0.56	0.64	0.68	0.70	0.72	0.76	0.76	0.78	0.78	0.82	0.78	0.72	0.70
29	0.63	0.71	0.75	0.78	0.79	0.84	0.84	0.86	0.86	0.90	0.86	0.80	0.78
30	0.70	0.78	0.82	0.87	0.87	0.92	0.92	0.94	0.94	0.98	0.94	0.88	0.86
35	1.10	1.17	1.22	1.24	1.30	1.32	1.33	1.35	1.36	1.39	1.34	1.27	1.25
40	1.50	1.61	1.67	1.71	1.73	1.79	1.79	1.80	1.82	1.83	1.78	1.69	1.65
50	2.65	2.71	2.74	2.78	2.80	2.80	2.80	2.80	2.79	2.70	2.56	2.51
60	3.87	3.88	3.88	3.88	3.88	3.88	3.88	3.90	3.82	3.70	3.43	3.41
70	5.17	5.18	5.20	5.14	5.13	5.10	5.08	5.06	4.90	4.72	4.47	4.35
80	6.62	6.59	6.54	6.46	6.38	6.30	6.26	6.06	5.82	5.50	5.33
90	8.26	8.16	8.06	7.97	7.83	7.71	7.58	7.30	6.96	6.58	6.37
100	10.01	9.87	9.72	9.56	9.39	9.21	9.03	8.64	8.22	7.76	7.42

Example.—A sugar solution shows a reading of 30.2° Brix at 30°C. To find the necessary correction for the conversion of this reading to the reading which would have been obtained if the observation had been made at 17.5°C., find the vertical column in the table headed 30° Brix, which is the nearest to the observed reading. Follow down this column until the number is reached which is opposite to the temperature of observation—in this case 30°. The number found, 0.92, is to be added to the observed reading.

7

By Means of a Pycnometer.—Official.

(a) *By specific gravity at $\frac{20^\circ\text{C.}}{4^\circ}$.*—Determine the specific gravity of the solution at $\frac{20^\circ\text{C.}}{4^\circ}$ by means of a pycnometer and ascertain the corresponding per cent by weight of sucrose from 9. When the density of the substance is too high for a direct determination, dilute and calculate the sucrose content of the original material as directed under 5.

(b) *By specific gravity at $\frac{17.5^\circ\text{C.}}{17.5^\circ}$.*—Proceed as directed under (a), the determinations of specific gravity being made at $\frac{17.5^\circ\text{C.}}{17.5^\circ}$ instead of at $\frac{20^\circ\text{C.}}{4^\circ}$. Ascertain the corresponding per cent by weight of sucrose from 8.

The pycnometer determination should not be made at any other temperature than $\frac{17.5^\circ\text{C.}}{17.5^\circ}$ or $\frac{20^\circ\text{C.}}{4^\circ}$.

8

TABLE 10.

For the comparison of specific gravities at $\frac{17.5^{\circ}\text{C.}}{17.5^{\circ}}$, degrees Brix and degrees Baumé.

$$\text{Degree Baumé} = 146.78 - \frac{146.78}{\text{sp. gr.}}$$

DEGREE BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY	DEGREE BAUMÉ	DEGREE BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY	DEGREE BAUMÉ	DEGREE BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY	DEGREE BAUMÉ
1.0	1.00388	0.6	33.0	1.14423	18.5	65.0	1.31989	35.6
2.0	1.00779	1.1	34.0	1.14915	19.05	66.0	1.32601	36.1
3.0	1.01173	1.7	35.0	1.15411	19.6	67.0	1.33217	36.6
4.0	1.01570	2.3	36.0	1.15911	20.1	68.0	1.33836	37.1
5.0	1.01970	2.8	37.0	1.16413	20.7	69.0	1.34460	37.6
6.0	1.02373	3.4	38.0	1.16920	21.2	70.0	1.35088	38.1
7.0	1.02779	4.0	39.0	1.17430	21.8	71.0	1.35720	38.6
8.0	1.03187	4.5	40.0	1.17943	22.3	72.0	1.36355	39.1
9.0	1.03599	5.1	41.0	1.18460	22.9	73.0	1.36993	39.6
10.0	1.04014	5.7	42.0	1.18981	23.4	74.0	1.37639	40.1
11.0	1.04431	6.2	43.0	1.19505	23.95	75.0	1.38287	40.6
12.0	1.04852	6.8	44.0	1.20033	24.5	76.0	1.38939	41.1
13.0	1.05276	7.4	45.0	1.20565	25.0	77.0	1.39595	41.6
14.0	1.05703	7.9	46.0	1.21100	25.6	78.0	1.40254	42.1
15.0	1.06133	8.5	47.0	1.21639	26.1	79.0	1.40918	42.6
16.0	1.06566	9.0	48.0	1.22182	26.6	80.0	1.41586	43.1
17.0	1.07002	9.6	49.0	1.22728	27.2	81.0	1.42258	43.6
18.0	1.07441	10.1	50.0	1.23278	27.7	82.0	1.42934	44.1
19.0	1.07884	10.7	51.0	1.23832	28.2	83.0	1.43614	44.6
20.0	1.08329	11.3	52.0	1.24390	28.8	84.0	1.44298	45.1
21.0	1.08778	11.8	53.0	1.24951	29.3	85.0	1.44986	45.5
22.0	1.09231	12.4	54.0	1.25517	29.8	86.0	1.45678	46.0
23.0	1.09686	13.0	55.0	1.26086	30.4	87.0	1.46374	46.5
24.0	1.10145	13.5	56.0	1.26658	30.9	88.0	1.47074	47.0
25.0	1.10607	14.1	57.0	1.27235	31.4	89.0	1.47778	47.45
26.0	1.11072	14.6	58.0	1.27816	31.9	90.0	1.48486	47.9
27.0	1.11541	15.2	59.0	1.28400	32.5	91.0	1.49199	48.5
28.0	1.12013	15.7	60.0	1.28989	33.0	92.0	1.49915	48.9
29.0	1.12488	16.3	61.0	1.29581	33.5	93.0	1.50635	49.4
30.0	1.12967	16.8	62.0	1.30177	34.0	94.0	1.51359	49.8
31.0	1.13449	17.4	63.0	1.30777	34.5	95.0	1.52087	50.3
32.0	1.13934	17.95	64.0	1.31381	35.1			

When the number expressing the specific gravity found by analysis falls between the numbers given in the above table, the exact equivalent in degrees Brix or Baumé is found by a simple calculation.

Example.—The pycnometer shows the specific gravity of a certain sirup to be 1.20909. The table shows that the corresponding degree Brix is between 45.0 and 46.0. Subtracting the specific gravity of a solution of 45° Brix from the corresponding figure for 46°, we have (expressing the specific gravities as whole numbers) $121,100 - 120,565 = 535$, the difference in specific gravity for 1° Brix at this point in the table. Subtracting the specific gravity corresponding to 45° from the specific gravity found by analysis, we have $120,909 - 120,565 = 344$; $\frac{344}{535} = 0.64$, the fraction of 1° Brix more than 45°. The degree Brix, corresponding to a sp. gr. of 1.20909, is therefore 45.64.

TABLE 11.

Densities^a of solutions of cane sugar at 20°C.

(This table is the basis for standardizing hydrometers indicating per cent of sugar at 20°C.)

PER CENT SUGAR	TENTHS OF PER CENT									
	0	1	2	3	4	5	6	7	8	9
0	0.998324	0.998323	0.999010	0.999398	0.999786	1.000174	1.000563	1.000952	1.001342	1.001731
1	1.002120	1.002509	1.002897	1.003286	1.003675	1.004064	1.004453	1.004844	1.005234	1.005624
2	1.006015	1.006405	1.006796	1.007188	1.007580	1.007972	1.008363	1.008755	1.009148	1.009541
3	1.009934	1.010327	1.010721	1.011115	1.011510	1.011904	1.012298	1.012694	1.013089	1.013485
4	1.013881	1.014277	1.014673	1.015070	1.015467	1.015864	1.016261	1.016659	1.017058	1.017456
5	1.017854	1.018253	1.018652	1.019052	1.019451	1.019851	1.020251	1.020651	1.021053	1.021454
6	1.021855	1.022257	1.022659	1.023061	1.023463	1.023867	1.024270	1.024673	1.025077	1.025481
7	1.025885	1.026289	1.026694	1.027099	1.027504	1.027910	1.028316	1.028722	1.029128	1.029535
8	1.029942	1.030349	1.030757	1.031165	1.031573	1.031982	1.032391	1.032800	1.033209	1.033619
9	1.034029	1.034439	1.034850	1.035260	1.035671	1.036082	1.036494	1.036906	1.037318	1.037730
10	1.038143	1.038556	1.038970	1.039383	1.039797	1.040212	1.040628	1.041041	1.041456	1.041872
11	1.042288	1.042704	1.043121	1.043537	1.043954	1.044370	1.044788	1.045206	1.045625	1.046043
12	1.046462	1.046881	1.047300	1.047720	1.048140	1.048559	1.048980	1.049401	1.049822	1.050243
13	1.050665	1.051087	1.051510	1.051933	1.052356	1.052778	1.053202	1.053626	1.054050	1.054475
14	1.054900	1.055325	1.055750	1.056176	1.056602	1.057029	1.057455	1.057881	1.058310	1.058737
15	1.059165	1.059593	1.060022	1.060451	1.060880	1.061308	1.061738	1.062168	1.062598	1.063029
16	1.063460	1.063892	1.064324	1.064756	1.065188	1.065621	1.066054	1.066487	1.066921	1.067355
17	1.067789	1.068223	1.068658	1.069093	1.069529	1.069964	1.070400	1.070836	1.071273	1.071710
18	1.072147	1.072585	1.073023	1.073461	1.073900	1.074338	1.074777	1.075217	1.075657	1.076097
19	1.076537	1.076978	1.077419	1.077860	1.078302	1.078744	1.079187	1.079630	1.080072	1.080515
20	1.080959	1.081403	1.081848	1.082292	1.082737	1.083182	1.083628	1.084074	1.084520	1.084967
21	1.085414	1.085861	1.086309	1.086757	1.087205	1.087652	1.088101	1.088550	1.089000	1.089450
22	1.089900	1.090351	1.090802	1.091253	1.091704	1.092155	1.092607	1.093060	1.093513	1.093966
23	1.094420	1.094874	1.095328	1.095782	1.096236	1.096691	1.097147	1.097603	1.098058	1.098514
24	1.098971	1.099428	1.099886	1.100344	1.100802	1.101259	1.101718	1.102177	1.102637	1.103097
25	1.103557	1.104017	1.104478	1.104938	1.105400	1.105862	1.106324	1.106786	1.107248	1.107711
26	1.108175	1.108639	1.109103	1.109568	1.110033	1.110497	1.110963	1.111429	1.111895	1.112361
27	1.112828	1.113295	1.113763	1.114239	1.114707	1.115185	1.115663	1.116140	1.116627	1.117104
28	1.117512	1.117982	1.118453	1.118923	1.119395	1.119867	1.120339	1.120812	1.121284	1.121757
29	1.122231	1.122705	1.123179	1.123653	1.124128	1.124603	1.125079	1.125555	1.126030	1.126507
30	1.126984	1.127461	1.127939	1.128417	1.128896	1.129374	1.129853	1.130332	1.130812	1.131292
31	1.131773	1.132254	1.132735	1.133216	1.133698	1.134180	1.134663	1.135146	1.135628	1.136112
32	1.136596	1.137080	1.137565	1.138049	1.138534	1.139020	1.139506	1.140000	1.140479	1.140966
33	1.141453	1.141941	1.142429	1.142916	1.143405	1.143894	1.144384	1.144874	1.145363	1.145854
34	1.146345	1.146836	1.147328	1.147820	1.148313	1.148806	1.149298	1.149792	1.150286	1.150780
35	1.151275	1.151770	1.152265	1.152760	1.153256	1.153752	1.154249	1.154746	1.155242	1.155740
36	1.156238	1.156738	1.157235	1.157733	1.158233	1.158733	1.159233	1.159733	1.160233	1.160734
37	1.161236	1.161738	1.162240	1.162742	1.163245	1.163748	1.164252	1.164756	1.165259	1.165764
38	1.166269	1.166775	1.167281	1.167786	1.168293	1.168800	1.169307	1.169815	1.170322	1.170831
39	1.171340	1.171849	1.172359	1.172869	1.173379	1.173889	1.174400	1.174911	1.175423	1.175935
40	1.176447	1.176960	1.177473	1.177987	1.178501	1.179014	1.179527	1.180044	1.180560	1.181076
41	1.181592	1.182108	1.182625	1.183142	1.183660	1.184178	1.184696	1.185215	1.185734	1.186253
42	1.186773	1.187293	1.187814	1.188335	1.188856	1.189379	1.189901	1.190423	1.190946	1.191469
43	1.191993	1.192517	1.193041	1.193565	1.194090	1.194616	1.195141	1.195667	1.196193	1.196720
44	1.197247	1.197775	1.198303	1.198832	1.199360	1.199890	1.200420	1.200950	1.201480	1.202010
45	1.202540	1.203071	1.203603	1.204136	1.204668	1.205200	1.205733	1.206266	1.206801	1.207335
46	1.207870	1.208405	1.208940	1.209477	1.210013	1.210549	1.211086	1.211623	1.212162	1.212700
47	1.213238	1.213777	1.214315	1.214856	1.215395	1.215936	1.216476	1.217017	1.217559	1.218101
48	1.218643	1.219185	1.219729	1.220272	1.220815	1.221360	1.221904	1.222449	1.222995	1.223540
49	1.224086	1.224632	1.225180	1.225727	1.226274	1.226823	1.227371	1.227919	1.228469	1.229018
50	1.229567	1.230117	1.230668	1.231219	1.231770	1.232322	1.232874	1.233426	1.233979	1.234532
51	1.235085	1.235639	1.236194	1.236748	1.237303	1.237859	1.238414	1.238970	1.239527	1.240084
52	1.240641	1.241198	1.241757	1.242315	1.242873	1.243433	1.243992	1.244552	1.245113	1.245673
53	1.246234	1.246795	1.247358	1.247920	1.248482	1.249046	1.249609	1.250172	1.250737	1.251301
54	1.251866	1.252431	1.252997	1.253563	1.254129	1.254697	1.255264	1.255831	1.256400	1.256967
55	1.257535	1.258104	1.258674	1.259244	1.259815	1.260385	1.260955	1.261527	1.262099	1.262671
56	1.263245	1.263816	1.264390	1.264963	1.265537	1.266112	1.266688	1.267261	1.267837	1.268413
57	1.268989	1.269565	1.270143	1.270720	1.271299	1.271877	1.272455	1.273035	1.273614	1.274194
58	1.274774	1.275354	1.275936	1.276517	1.277098	1.277680	1.278262	1.278844	1.279428	1.280011
59	1.280595	1.281179	1.281764	1.282349	1.282935	1.283521	1.284107	1.284694	1.285281	1.285869

TABLE 11.—Continued.
Densities of solutions of cane sugar at 20°C.

PER CENT SUGAR	TENTHS OF PER CENT								
	0	1	2	3	4	5	6	7	8
60	1.286456	1.287044	1.287633	1.288222	1.288811	1.289401	1.289991	1.290581	1.291172
61	1.292354	1.292946	1.293539	1.294131	1.294725	1.295318	1.295911	1.296506	1.297099
62	1.298991	1.299586	1.299483	1.300079	1.300677	1.301274	1.301871	1.302470	1.303068
63	1.304267	1.304867	1.305467	1.306068	1.306669	1.307271	1.307872	1.308475	1.309077
64	1.310282	1.310885	1.311489	1.312093	1.312699	1.313304	1.313909	1.314516	1.315121
65	1.316334	1.316941	1.317549	1.318157	1.318766	1.319374	1.319983	1.320593	1.321203
66	1.322425	1.323036	1.323648	1.324259	1.324872	1.325484	1.326097	1.326711	1.327325
67	1.328554	1.329170	1.329785	1.330401	1.331017	1.331633	1.332250	1.332868	1.333485
68	1.334722	1.335342	1.335961	1.336581	1.337200	1.337821	1.338441	1.339063	1.339684
69	1.340928	1.341551	1.342174	1.342798	1.343421	1.344046	1.344671	1.345296	1.345922
70	1.347174	1.347801	1.348427	1.349055	1.349682	1.350311	1.350939	1.351568	1.352197
71	1.353456	1.354087	1.354717	1.355349	1.355980	1.356612	1.357245	1.357877	1.358511
72	1.359778	1.360413	1.361047	1.361682	1.362317	1.362953	1.363589	1.364226	1.364864
73	1.366139	1.366777	1.367415	1.368054	1.368693	1.369333	1.369973	1.370613	1.371254
74	1.372536	1.373178	1.373820	1.374463	1.375105	1.375749	1.376392	1.377036	1.377680
75	1.379071	1.379617	1.380262	1.380909	1.381555	1.382203	1.382851	1.383499	1.384148
76	1.385446	1.386096	1.386745	1.387396	1.388046	1.388696	1.389347	1.389997	1.390651
77	1.391956	1.392610	1.393263	1.393917	1.394571	1.395226	1.395881	1.396536	1.397192
78	1.398505	1.399162	1.399819	1.400477	1.401134	1.401793	1.402452	1.403111	1.403771
79	1.405091	1.405753	1.406412	1.407074	1.407735	1.408398	1.409061	1.409723	1.410387
80	1.411715	1.412380	1.413044	1.413709	1.414374	1.415040	1.415706	1.416373	1.417039
81	1.418374	1.419043	1.419711	1.420380	1.421049	1.421719	1.422390	1.423060	1.423730
82	1.425072	1.425744	1.426416	1.427089	1.427761	1.428435	1.429109	1.429782	1.430457
83	1.431807	1.432483	1.433158	1.433835	1.434511	1.435188	1.435866	1.436543	1.437222
84	1.438579	1.439259	1.439938	1.440619	1.441299	1.441980	1.442661	1.443342	1.444024
85	1.445388	1.446071	1.446754	1.447438	1.448121	1.448806	1.449491	1.450175	1.450860
86	1.452232	1.452919	1.453606	1.454292	1.454980	1.455668	1.456357	1.457045	1.457735
87	1.459114	1.459805	1.460496	1.461186	1.461877	1.462568	1.463260	1.463953	1.464645
88	1.466032	1.466726	1.467420	1.468115	1.468810	1.469506	1.470200	1.470896	1.471592
89	1.472966	1.473664	1.474361	1.475060	1.475759	1.476457	1.477156	1.477855	1.478555
90	1.479976	1.480677	1.481378	1.482080	1.482782	1.483484	1.484187	1.484890	1.485593
91	1.487002	1.487707	1.488411	1.489117	1.489823	1.490528	1.491234	1.491941	1.492647
92	1.494063	1.494771	1.495479	1.496188	1.496897	1.497606	1.498316	1.499026	1.499736
93	1.501158	1.501870	1.502582	1.503293	1.504006	1.504719	1.505432	1.506146	1.506859
94	1.508259	1.508904	1.509720	1.510435	1.511151	1.511868	1.512585	1.513303	1.514019
95	1.515455	1.516174	1.516893	1.517612	1.518332	1.519051	1.519771	1.520492	1.521212
96	1.522556	1.523278	1.524000	1.524823	1.525546	1.526269	1.526993	1.527717	1.528441
97	1.529891	1.530616	1.531342	1.532068	1.532794	1.533521	1.534248	1.534976	1.535704
98	1.537161	1.537889	1.538618	1.539347	1.540076	1.540806	1.541536	1.542267	1.542998
99	1.544462	1.545194	1.545926	1.546659	1.547392	1.548127	1.548861	1.549596	1.550329
100	1.551800								1.551064

REFRACTOMETER METHOD.—TENTATIVE.

Determine the refractive index of the solution at 28°C. and obtain the corresponding percentage of dry substance from 11. If the refractive index is obtained at a temperature other than 28°C., correct the result as indicated in 12. If the solution is too dark to be read in the instrument, dilute with a concentrated sugar solution. Water should never be used for this purpose. Mix weighed amounts of the solution under examination and a solution of pure sugar of about the same strength, and obtain the amount of dry substance in the former by the following formula:

$$x = \frac{(A + B) C - BD}{A} \text{ in which}$$

x = per cent of dry substance to be found;

A = weight in grams of the material mixed with B;

B = weight in grams of pure sugar solution employed in the dilution;

C = per cent of dry substance in the mixture of A and B obtained from the refractive index;

D = per cent of dry substance in the pure sugar solution obtained from its refractive index.

11

TABLE 12.—GEERLIGS' TABLE.

For dry substance in sugar-house products by the Abbe refractometer, at 28°C.

INDEX	PER CENT DRY SUBSTANCE	DECIMALS TO BE ADDED FOR FRACTIONAL READINGS*	INDEX	PER CENT DRY SUBSTANCE	DECIMALS TO BE ADDED FOR FRACTIONAL READINGS*	INDEX	PER CENT DRY SUBSTANCE	DECIMALS TO BE ADDED FOR FRACTIONAL READINGS*
1.3335	1	0.0001 = 0.05	1.3484	11	0.0001 = 0.05	1.3746	27	0.0001 = 0.05
1.3349	2	0.0002 = 0.1	1.3500	12	0.0002 = 0.1	1.3764	28	0.0002 = 0.1
1.3364	3	0.0003 = 0.2	1.3516	13	0.0003 = 0.2	1.3782	29	0.0003 = 0.15
1.3379	4	0.0004 = 0.25	1.3530	14	0.0004 = 0.25	1.3800	30	0.0004 = 0.2
1.3394	5	0.0005 = 0.3	1.3546	15	0.0005 = 0.3	1.3818	31	0.0005 = 0.25
1.3409	6	0.0006 = 0.4	1.3562	16	0.0006 = 0.4	1.3836	32	0.0006 = 0.3
1.3424	7	0.0007 = 0.5	1.3578	17	0.0007 = 0.45	1.3854	33	0.0007 = 0.35
1.3439	8	0.0008 = 0.6	1.3594	18	0.0008 = 0.5	1.3872	34	0.0008 = 0.4
1.3454	9	0.0009 = 0.7	1.3611	19	0.0009 = 0.6	1.3890	35	0.0009 = 0.45
1.3469	10	0.0010 = 0.75	1.3627	20	0.0010 = 0.65	1.3909	36	0.0010 = 0.5
		0.0011 = 0.8	1.3644	21	0.0011 = 0.7	1.3928	37	0.0011 = 0.55
		0.0012 = 0.8	1.3661	22	0.0012 = 0.75	1.3947	38	0.0012 = 0.6
		0.0013 = 0.85	1.3678	23	0.0013 = 0.8	1.3966	39	0.0013 = 0.65
		0.0014 = 0.9	1.3695	24	0.0014 = 0.85	1.3984	40	0.0014 = 0.7
		0.0015 = 1.0	1.3712	25	0.0015 = 0.9	1.4003	41	0.015 = 0.75
			1.3729	26	0.0016 = 0.95			0.016 = 0.8
								0.017 = 0.85
								0.018 = 0.9
								0.019 = 0.95
								0.020 = 1.0
								0.021 = 1.0
1.4023	42	0.0001 = 0.05	1.4292	55	0.0001 = 0.05	1.4711	73	0.0001 = 0.0
1.4043	43	0.0002 = 0.1	1.4314	56	0.0002 = 0.1	1.4736	74	0.0002 = 0.05
1.4063	44	0.0003 = 0.15	1.4337	57	0.0003 = 0.1	1.4761	75	0.0003 = 0.1
1.4083	45	0.0004 = 0.2	1.4359	58	0.0004 = 0.15	1.4786	76	0.0004 = 0.15
1.4104	46	0.0005 = 0.25	1.4382	59	0.0005 = 0.2	1.4811	77	0.0005 = 0.2
1.4124	47	0.0006 = 0.3	1.4405	60	0.0006 = 0.25	1.4836	78	0.0006 = 0.25
1.4145	48	0.0007 = 0.35	1.4428	61	0.0007 = 0.3	1.4862	79	0.0007 = 0.3
1.4166	49	0.0008 = 0.4	1.4451	62	0.0008 = 0.35	1.4888	80	0.0008 = 0.35
1.4186	50	0.0009 = 0.45	1.4474	63	0.0009 = 0.4	1.4914	81	0.0009 = 0.4
1.4207	51	0.0010 = 0.5	1.4497	64	0.0010 = 0.45	1.4940	82	0.0010 = 0.45
1.4228	52	0.0011 = 0.55	1.4520	65	0.0011 = 0.5	1.4966	83	0.0011 = 0.5
1.4249	53	0.0012 = 0.6	1.4543	66	0.0012 = 0.55	1.4992	84	0.0012 = 0.55
1.4270	54	0.0013 = 0.65	1.4567	67	0.0013 = 0.6	1.5019	85	0.0013 = 0.6
		0.0014 = 0.7	1.4591	68	0.0014 = 0.65	1.5046	86	0.0014 = 0.65
		0.0015 = 0.75	1.4615	69	0.0015 = 0.7	1.5073	87	0.0015 = 0.7
		0.0016 = 0.8	1.4639	70	0.0016 = 0.75	1.5100	88	0.0016 = 0.75
		0.0017 = 0.85	1.4663	71	0.0017 = 0.8	1.5127	89	0.0017 = 0.8
		0.0018 = 0.9	1.4687	72	0.0018 = 0.85	1.5155	90	0.0018 = 0.85
		0.0019 = 0.95			0.0019 = 0.9			0.0019 = 0.9
		0.0020 = 1.0			0.0020 = 0.95			0.0020 = 0.95
		0.0021 = 1.0			0.0021 = 1.0			0.0021 = 1.0
					0.0022 = 0.95			0.0022 = 0.95
					0.0023 = 1.0			0.0023 = 1.0
					0.0024 = 1.0			0.0024 = 1.0
								0.0025 = 0.9
								0.0025 = 0.95
								0.0026 = 1.0
								0.0027 = 1.0
								0.0028 = 1.0

* Find in the table the refractive index which is next lower than the reading actually made and note the corresponding whole number for the per cent of dry substance. Subtract the refractive index obtained from the table from the observed reading; the decimal corresponding to this difference, as given in the column so marked, is added to the whole per cent of dry substance as first obtained.

12

TABLE 13.
Corrections for temperature.

TEMPERATURE OF THE PRISMS IN °C.	DRY SUBSTANCE												
	0	5	10	15	20	25	30	40	50	60	70	80	90
	Subtract—												
20	0.53	0.54	0.55	0.56	0.57	0.58	0.60	0.62	0.64	0.62	0.61	0.60	0.58
21	0.46	0.47	0.48	0.49	0.50	0.51	0.52	0.54	0.56	0.54	0.53	0.52	0.50
22	0.40	0.41	0.42	0.42	0.43	0.44	0.45	0.47	0.48	0.47	0.46	0.45	0.44
23	0.33	0.33	0.34	0.35	0.36	0.37	0.38	0.39	0.40	0.39	0.38	0.38	0.38
24	0.26	0.26	0.27	0.28	0.28	0.29	0.30	0.31	0.32	0.31	0.31	0.30	0.30
25	0.20	0.20	0.21	0.21	0.22	0.22	0.23	0.23	0.24	0.23	0.23	0.23	0.22
26	0.12	0.12	0.13	0.14	0.14	0.15	0.15	0.16	0.16	0.16	0.15	0.15	0.14
27	0.07	0.07	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.07
	Add—												
29	0.07	0.07	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.07
30	0.12	0.12	0.13	0.14	0.14	0.14	0.15	0.15	0.16	0.16	0.16	0.15	0.14
31	0.20	0.20	0.21	0.21	0.22	0.22	0.23	0.23	0.24	0.23	0.23	0.23	0.22
32	0.26	0.26	0.27	0.28	0.28	0.29	0.30	0.31	0.32	0.31	0.31	0.30	0.30
33	0.33	0.33	0.34	0.35	0.36	0.37	0.38	0.39	0.40	0.39	0.38	0.38	0.38
34	0.40	0.41	0.42	0.42	0.43	0.44	0.45	0.47	0.48	0.47	0.46	0.45	0.44
35	0.46	0.47	0.48	0.49	0.50	0.51	0.52	0.54	0.56	0.54	0.53	0.52	0.50

ASH.

13

Method I.—Official.

Heat 5–10 grams of the sample in a 50–100 cc. platinum dish at 100°C. until the water is expelled, add a few drops of pure olive oil, and heat slowly over a flame until swelling ceases. Then place the dish in a muffle and heat at low redness until a white ash is obtained.

14

Method II.—Official.

Carbonize the mass at a low heat, dissolve the soluble salts in hot water, burn the residual mass as directed in 13, add the solution of soluble salts, and evaporate to dryness at 100°C., ignite gently, cool in a desiccator, and weigh.

15

Method III.—Official.

Saturate the sample with sulphuric acid, dry, ignite gently, then burn in a muffle at low redness. Deduct one tenth of the weight of the ash, and calculate the per cent.

16

QUANTITATIVE ANALYSIS OF THE ASH.—OFFICIAL.

Proceed as directed under III.

17

SOLUBLE AND INSOLUBLE ASH.—TENTATIVE.

Ash the material as directed under 13 or 14. Add water to the ash in the platinum dish, heat nearly to boiling, filter through an ashless filter paper, and wash with hot water until the combined filtrate and washings measure about 60 cc. Re

turn the filter paper and contents to the platinum dish, ignite carefully, and weigh. Calculate the percentages of water-soluble and water-insoluble ash.

18 ALKALINITY OF THE SOLUBLE ASH.—TENTATIVE.

Cool the filtrate from 17 and titrate with N/10 hydrochloric acid, using methyl orange as an indicator.

Express the alkalinity in terms of the number of cc. of N/10 acid per 1 gram of the sample.

19 ALKALINITY OF THE INSOLUBLE ASH.—TENTATIVE.

Add an excess of N/10 hydrochloric acid (usually 10–15 cc.) to the ignited insoluble ash in the platinum dish, under 17, heat to boiling over an asbestos plate, cool, and titrate the excess of hydrochloric acid with N/10 sodium hydroxid, using methyl orange as an indicator.

Express the alkalinity in terms of the number of cc. of N/10 acid per 1 gram of the sample.

20 MINERAL ADULTERANTS IN THE ASH.—TENTATIVE.

Mix 100 grams of molasses, sirup, honey, or the confectionery solution prepared as directed under 1 (b) and evaporate to a sirupy consistency, with about 35 grams of concentrated sulphuric acid in a large porcelain evaporating dish. Pass an electric current through it while stirring by placing one platinum electrode in the bottom of the dish near one side and attaching the other to the lower end of the glass rod with which the contents are stirred. Begin with a current of about 1 ampere and gradually increase to 4 (modified from method of Budde and Schou* for determining nitrogen electrolytically). In 10–15 minutes the mass is reduced to a fine dry char, which may be readily burnt to a white ash in the original dish over a free flame or in a muffle.

This method* is preferred to the ordinary method of heating with sulphuric acid, especially in the case of molasses, because, if properly manipulated, it comes quietly into the form of a very finely divided char or powder, especially adapted for subsequent quick ignition.

If an electric current is not available, treat in a large porcelain dish 100 grams of the saccharine solution, evaporated to a sirupy consistency, with sufficient concentrated sulphuric acid to thoroughly carbonize the mass and ignite in the usual manner.

The following adulterants may be present: salts of tin, used in molasses to bleach; mineral pigments, such as chromate of lead in yellow confectionery; oxid of iron, sometimes used to simulate the color of chocolate; and copper. These elements may be detected by the usual qualitative tests.

21 NITROGEN.—TENTATIVE.

Determine nitrogen in 5 grams of the material as directed under I, 18, 21 or 23, using a larger quantity of the sulphuric acid if necessary for complete digestion.

SUCROSE.

22 Method I.—Tentative.

(Substances in which the volume of the combined insoluble matter and precipitate from clarifying agents is less than 1 cc. from 26 grams.)

Determine sucrose by polarisation before and after inversion, as directed under VIII, 14.

All products which contain dextrose or other reducing sugars in the crystalline form, or in supersaturated solution, exhibit the phenomenon of birotation. The constant rotation only should be employed in the Clerget formula, and to obtain this the solutions prepared for direct polarisation should be allowed to stand overnight before making the reading. If it is desired to make the direct reading immediately, the birotation may be destroyed by heating the neutral solution to boiling for a few minutes or by adding a few drops of strong ammonium hydroxid before completing the volume.

23

Method II. (Double dilution method.⁶)—Tentative.

(Substances in which the volume of the combined insoluble matter and precipitate from clarifying agents is more than 1 cc. from 26 grams.)

Weigh out a half normal weight of the sample and make up the solution to 100 cc., employing the appropriate clarifier (basic lead acetate for dark colored confectionery or molasses and alumina cream for light colored confectionery). Also weigh out a normal weight of the sample and make up a second solution with the clarifier to 100 cc. Filter and obtain direct polariscopic readings of both solutions. Invert each solution as directed in 22 and obtain its invert reading.

The true direct polarisation of the sample is the product of the two direct readings divided by their difference.

The true invert polarisation is the product of the two invert readings divided by their difference.

Calculate the sucrose from the true polarizations thus obtained by the formula given under VIII, 14.

COMMERCIAL GLUCOSE (APPROXIMATE).

24

Method I.—Tentative.

(Substances containing little or no invert sugar.)

Commercial glucose can not be determined accurately owing to the varying amounts of dextrin, maltose, and dextrose present in this product. However, in sirups, in which the amount of invert sugar is so small as not to appreciably affect the result, commercial glucose may be estimated approximately by the following formula:⁷

$$G = \frac{(a - S) 100}{175} \text{ in which}$$

G = per cent of commercial glucose;

a = direct polarisation;

S = per cent of cane sugar.

Express the results in terms of commercial glucose polarising +175°V.

Method II.—Tentative.

25

(Substances containing invert sugar.⁷)

Prepare an inverted half normal solution of the substance as directed under VIII, 14 except that after inversion cool the solution, make neutral to phenolphthalein with sodium hydroxid solution, slightly acidify with hydrochloric acid, and treat with 5-10 cc. of alumina cream before making up to the mark. Filter and polarize at 87°C. in a 200 mm. jacketed tube. Multiply the reading by 200 and divide by the factor 163 to express the amount of glucose present in terms of glucose polarizing +175°V.

26

REDUCING SUGARS.—TENTATIVE.

Determine either as dextrose or invert sugar as directed under VIII, 50, 51, 52, 54, or 21, 23, 25, 36 or 39.

27

STARCH.—TENTATIVE.

Measure 25 cc. of a solution or uniform mixture, prepared as directed in 1 (b), (representing 5 grams of the sample) into a 300 cc. beaker, or introduce 5 grams of the finely ground sample (previously extracted with ether if the sample contains much fat) into the beaker, add sufficient water to make the volume 100 cc., heat to about 60°C. (avoiding if possible gelatinizing the starch) and allow to stand for about an hour, stirring frequently to secure complete solution of the sugars. Transfer to a stout wide-mouthed bottle, rinse the beaker with a little warm water, cool, add an equal volume of 95% alcohol, mix, and allow to stand at least an hour. Centrifugalize until the precipitate is closely packed on the bottom of the bottle and decant the supernatant liquid through a hardened filter. Wash the precipitate with successive 50 cc. portions of 50% alcohol by centrifugalizing and decanting through the filter until 3 or 4 drops of the washings give no test for sugar with alphanaphthol as described under 68. Transfer the residue from the bottle and the hardened filter to a large flask and determine starch as directed under VIII, 60.

ETHER EXTRACT IN CONFECTIONERY.

28

Continuous Extraction.—Tentative.

(1) Measure 25 cc. of a 20% mixture or solution, prepared as directed under 1 (b), into a very thin, readily frangible, glass evaporating shell (*Hofmeister Schälchen*), containing 5-7 grams of freshly ignited asbestos fiber; or (2) If impossible to obtain a uniform sample, weigh 5 grams of the mixed finely divided sample into a dish, and wash with water upon the asbestos in the evaporating shell, using, if necessary, a small portion of the asbestos fiber on a stirring rod to transfer the last traces of the sample from the dish to the shell. Dry to constant weight at 100°C., cool, wrap loosely in smooth paper, crush into rather small fragments between the fingers, transfer carefully the crushed mass, exclusive of the paper, to an extraction tube or a fat extraction cartridge. A thin lead disk (bottle cap) may be substituted for the *Schälchen*. The disk may then be cut into small pieces and placed in the extraction tube. Extract with anhydrous ether or petroleum ether (b. p. 45°-60°C. and without weighable residue) in a continuous extraction apparatus for at least 25 hours. In most cases it is advisable to remove the substance from the extractor after the first 12 hours, grind with sand to a fine powder, and re-extract for the remaining 13 hours. Transfer the extract to a tared flask, evaporate the solvent, dry to constant weight in an oven at 100°C.

29

Roese-Gottlieb Method.—Tentative.

Substances such as butter-scotch, invariably yield extremely inaccurate results by the above method. In such cases introduce 4 grams of the material, or an amount of a uniform solution equivalent to this amount of the dry substance, into a Röhrig tube or similar apparatus, make up to a volume of 10 cc. with water, add 1.25 cc. of concentrated ammonium hydroxid and mix thoroughly. Add 10 cc. of 95% alcohol and mix. Then add 25 cc. of washed ether and shake vigorously for half a minute; then add 25 cc. of petroleum ether (b. p. below 60°C.), and shake again for half a minute. Allow to stand for 20 minutes or until the separation between the

liquids is complete. Draw off as much as possible of the ether-fat solution (usually 0.5-0.8 cc. will be left) into a weighed flask through a small, rapid filter. The flask should be weighed with a similar one as a counterpoise. Again extract the liquid remaining in the tube, this time with 15 cc. each of ether and petroleum ether, shake vigorously half a minute with each, and allow to settle. Proceed as above, washing the tip of the spigot and the filter with a few cc. of a mixture of equal parts of the 2 ethers (previously mixed and free from deposited water). For absolutely exact results the extraction must be repeated. This third extraction usually yields not more than about 1 mg. of fat, if the previous ether-fat solutions have been drawn off closely, or an amount averaging about 0.02% on a 4 gram charge. Evaporate the ether slowly on a steam bath, then dry the fat in a boiling water oven until the loss in weight ceases. Test the purity of the fat by dissolving in a little petroleum ether. Should a residue remain, wash the fat out completely with petroleum ether, dry the residue, weigh, and deduct the weight.

30**PARAFFIN IN CONFECTIONERY.—TENTATIVE.**

Add to the ether extract in the flask, as above obtained, 10 cc. of 95% alcohol and 2 cc. of sodium hydroxid solution (1 to 1), connect the flask with a reflux condenser, and heat for an hour on the water bath, or until saponification is complete. Remove the condenser and allow the flask to remain on the bath until the alcohol is evaporated and the residue is dry. Dissolve the residue as completely as possible in about 40 cc. of water and heat on the bath, shaking frequently. Wash into a separatory funnel, cool, and extract with 4 successive portions of petroleum ether, which are collected in a tared flask or capsule. Evaporate the petroleum ether and dry in the oven to constant weight.

Any phytosterol or cholesterol present in the fat would be extracted with the paraffin. The amount is so insignificant that it may be disregarded generally. The character of the final residue should, however, be confirmed by determining its melting point, specific gravity, and refractive index.

31 ALCOHOL IN SIRUPS USED IN CONFECTIONERY ("BRANDY DROPS").—TENTATIVE.

Collect in a beaker the sirup from a sufficient number of pieces to yield 30-50 grams of sirup. Strain the sirup into a tared beaker and weigh. Introduce the sirup into a 250-300 cc. distilling flask, dilute with half its volume of water, attach the flask to a vertical condenser and distil almost 50 cc., or as much of the liquid as possible without causing charring. Foaming may be prevented by adding a little tannin, or a piece of paraffin about the size of a pea, to the contents of the distillation flask. Cool the distillate, make up to volume with water, mix well, and ascertain the specific gravity of the liquid by means of a pycnometer, and obtain the corresponding weight of alcohol in the 50 cc. of distillate from **XVI, 5**. Calculate the per cent by weight of alcohol in the candy filling.

32**COLORING MATTER.—TENTATIVE.**

Proceed as directed under **XI**.

33**METALS.—TENTATIVE.**

Proceed as directed under **XII**.

HONEY.*

34

PREPARATION OF SAMPLE.—TENTATIVE.

(a) *Liquid or strained honey*.—If the sample is free from granulation, mix thoroughly by stirring or shaking before drawing weighed portions for the analytical determination. If the honey is granulated, place the container, having the stopper loose, in a water bath, and heat at a temperature not exceeding 50°C. until the sugar crystals dissolve; mix thoroughly, cool, and weigh portions for the analytical determinations. If sediment such as particles of comb, wax, sticks, bees, etc., are present, heat the sample to 40°C. in a water bath and filter through cheese-cloth before weighing portions for analysis.

(b) *Comb honey*.—Cut across the top of the comb, if sealed, and separate completely from the comb by straining through a 40 mesh sieve. When portions of the comb or wax pass through the sieve, heat the sample as in (a) and strain through cloth. If the honey is granulated in the comb, heat until the wax is liquified, stir, cool, remove the wax and take the clear liquid for analysis.

35

MOISTURE.

Weigh 2 grams of the sample into a tared, flat-bottomed aluminium dish, having a diameter of about 60 mm. and containing 10–15 grams of fine quartz sand, which has been previously washed, dried and ignited, and a small glass stirring rod; add 5–10 cc. of water and thoroughly incorporate with the sand and honey mixture by means of the rod; dry the dish and its contents to constant weight in a vacuum oven at a temperature not exceeding 70°C.

36

ASH.—OFFICIAL.

Weigh 5–10 grams of honey into a platinum dish, add a few drops of pure olive oil to prevent spattering, and heat carefully until swelling ceases and then ignite at a temperature not above dull redness until a white ash is obtained.

37

SOLUBLE ASH.—TENTATIVE.

Proceed as directed under 17.

38

ALKALINITY OF THE SOLUBLE ASH.—TENTATIVE.

Proceed as directed under 18.

POLARIZATION.

39

Direct Polarization.—Tentative.

(a) *Immediate direct polarization*.—Transfer 26 grams of the honey to a 100 cc. flask with water, add 5 cc. of alumina cream, dilute to the mark with water at 20°C., filter, and polarize immediately in a 200 mm. tube.

(b) *Constant direct polarization*.—Pour the solution from the tube used in reading (a) back into the flask, stopper, and allow to stand for 24 hours. At the end of this time again polarize the solution at 20°C. in a 200 mm. tube.

(c) *Birotation*.—The difference between (a) and (b) gives the birotation.

(d) *Direct polarization at 87°C.*—Polarize the solution, obtained in (b), at 87°C. in a jacketed 200 mm. tube.

40

Invert Polarization.—Tentative.

(a) *At 20°C.*—Invert 50 cc. of the solution obtained in 39 as directed under VIII, 14 or 16, and polarize at 20°C. in a 200 mm. tube.

(b) *At 87°C.*—Polarize the solution, obtained as directed in (a), at 87°C. in a 200 mm. jacketed tube.

41

REDUCING SUGARS.—TENTATIVE.

Dilute 10 cc. of the solution, used for direct polarisation, 39, to 250 cc. and determine reducing sugars in 25 cc. of this solution by one of the methods given under VIII, 25, 36, 39 or 56, respectively. Calculate the result to per cent of invert sugar.

42

SUCROSE.—TENTATIVE.

Proceed as directed under VIII, 18. Determine reducing sugars after inversion by diluting 10 cc. of the solution obtained in 40, with a small amount of water, neutralizing with sodium carbonate, and making up to 250 cc. with water. Employ 50 cc. of this solution for the determination, using the same method as in 41.

43

LEVULOSE.—TENTATIVE.

Multiply the direct reading at 87°C., 39 (d), by 1.0315 and subtract the product from the constant direct polarization at 20°C., 39 (b); divide the difference by 2.3919 to obtain the grams of levulose in a normal weight of the honey. From this figure calculate the per cent of levulose in the original sample.

44

DEXTROSE.—TENTATIVE.

Subtract the per cent of levulose, obtained in 43, from the per cent of invert sugar, found in 41, to obtain the approximate per cent of dextrose.

The dextrose can be determined more accurately by multiplying the per cent of levulose, as found in 43, by the factor 0.915, which gives its dextrose equivalent in copper reducing power. Subtract this figure from that of the reducing sugars, 41, calculated as dextrose, to obtain the percentage of dextrose in the sample. (Owing to the difference in the reducing powers of different sugars, the sum of the dextrose thus found and the levulose as obtained in 43 will be greater than the amount of invert sugar obtained in 41).

45

DEXTRIN (APPROXIMATE).—TENTATIVE.

Transfer 8 grams of the sample (4 grams in the case of dark colored honey-dew honey) to a 100 cc. flask (using not more than 4 cc. of water) by allowing the sample to drain from the weighing dish into the flask and then dissolving the residue in 2 cc. of water. After adding this solution to the contents of the flask, rinse the weighing dish with two 1 cc. portions of water to which a little alcohol is added subsequently. Fill the flask to the mark with absolute alcohol, shaking constantly. Set the flask aside until the dextrin has collected on the sides and bottom and the liquid is clear. Decant the clear liquid through a filter paper and wash the residue in the flask with 10 cc. of 95% alcohol, pouring the washings through the same filter. Dissolve the dextrin in the flask with boiling water and filter through the filter paper already used, receiving the filtrate in a tared dish, prepared as directed under 4. Rinse the flask and wash the filter a number of times with small portions of hot water, evaporate on a water bath and dry to constant weight in vacuo at 70°C.

After determining the weight of the alcohol precipitate, dissolve the latter in water and make up to definite volume, using 50 cc. of water for each 0.5 gram of precipitate or part thereof.

Determine reducing sugars in the solution both before and after inversion as directed under VIII, 18, expressing the results as invert sugar. Calculate sucrose from the results thus obtained and subtract the sum of the reducing sugars before inversion and sucrose from the weight of the total alcoholic precipitate to obtain the weight of the dextrin.

46

FREE ACID.—TENTATIVE.

Dissolve 10 grams of the honey in water and titrate with N/10 sodium hydroxid using phenolphthalein as an indicator. Express the results in terms of cc. of N/10 sodium hydroxid required to neutralize 100 grams of the sample.

47

GLUCOSE.—TENTATIVE.

Qualitative test.—Dilute the honey with water in the proportion of 1 to 1, then add a few cc. of iodine solution (1 gram of iodine, 3 grams of potassium iodide, 50 cc. of water). In the presence of glucose the solution turns red or violet, the depth and character of the color depending upon the quality and nature of the glucose employed. A blank test with a pure honey of about the same color should be made in order to secure an accurate color comparison. Should the honey be dark and the percentage of glucose very small, precipitate the dextrin which may be present by adding several volumes of 95% alcohol. Allow to stand until the precipitate settles (do not filter), decant the liquid, dissolve the residue of dextrins in hot water, cool and apply the above test to this solution. A negative result is not proof of the absence of glucose as some glucose, especially of high conversion, does not give any reaction with iodine.⁹

Quantitative test.—An approximate determination can be made by Browne's formula as follows: Multiply the difference in the polarizations of the invert solution at 20°C. and 87°C. by 77 and divide this product by the percentage of invert sugar after inversion found in the sample. Multiply the quotient by 100 and divide the product by 26.7, to obtain the percentage of honey in the sample; 100 per cent minus the per cent of honey gives the percentage of glucose.

COMMERCIAL INVERT SUGAR.¹⁰

QUALITATIVE TESTS.

Fische Test (Bryan Modification¹¹).—Tentative.

48

REAGENT.

Resorcin solution.—Dissolve 1 gram of resorcin in 100 cc. of hydrochloric acid, sp. gr. 1.19.

49

MANIPULATION.

Introduce 10 cc. of a 50% honey solution into a test tube and add 5 cc. of ether. Shake gently and allow to stand for some time until the ether layer is clear. Transfer 2 cc. of this clear ether solution to a small test tube and add a large drop of the resorcin solution. Shake and note the color immediately. In the presence of artificial invert sugar, the resorcin assumes immediately an orange-red color turning to dark red.

*Feder Anilin Chlorid Test.*¹³—*Tentative.*

50

REAGENT.

Anilin chlorid solution.—To 100 cc. of C. P. anilin add 30 cc. of 25% hydrochloric acid.

51

MANIPULATION.

Introduce 5 grams of the honey into a porcelain dish and add 2.5 cc. of the anilin reagent. A bright red color indicates the presence of commercial invert sugar.

52

DIASTASE.¹⁴

Mix 1 part of honey with 2 parts of sterile water. Treat 10 cc. of this solution with 1 cc. of 1% soluble starch solution and digest at 45°C. for an hour. At the end of this time test the mixture with 1 cc. of iodine solution (1 gram of iodine, 2 grams of potassium iodide, 300 cc. of water). Treat another 10 cc. portion of the honey solution, mixed with 1 cc. of the soluble starch solution, without heating to 45°C., with the reagent and compare the colors produced. If the original honey had not been heated sufficiently to kill the diastase, an olive-green or brown coloration will be produced in the mixture that has been heated at 45°C. Heated or artificial honey becomes blue.

MAPLE PRODUCTS.

53

PREPARATION OF SAMPLE.—TENTATIVE.

(a) *Maple sirup.*—Determine the moisture by the method given under 54 (a). If the moisture is less than 35%, and there is some mineral sediment, pour the clear sirup into a beaker, washing the sediment also into the beaker with water. Then concentrate the sirup by boiling to a moisture content of about 35% (b. p. 104°C.). Set aside until cool, or preferably let the covered material stand overnight, and pour off the clear liquid for the analytical work. Where no sediment is present the sample is ready for analysis after careful mixing. Where sugar has crystallized out, warm to dissolve the sugar before starting the analysis. It is desirable in order to compare results upon different samples, to reduce all results other than moisture to a dry substance basis as determined in the clear sirup.

(b) *Maple sugar, maple cream, maple wax, etc.*—Determine moisture, by the method given under 54 (b), in the sample in its original condition by thoroughly mixing, if semi-plastic, or by rubbing up in a mortar representative portions of the product if solid. For all other analytical determinations use a solution prepared as follows: Weigh roughly 100 grams of the product into a beaker and dissolve by boiling with 200 cc. of water. Decant the resulting sirup while hot through a muslin filter, concentrate by boiling to a moisture content of 35% (b. p. 104°C.), cool, or preferably let the covered material stand overnight, set aside until clear, and use this clear sirup for analysis. It is desirable, in order to compare results upon different samples, that all results except moisture be expressed upon a dry basis.

54

MOISTURE.—TENTATIVE.

(a) *Maple sirup.*—Proceed as directed under 35 or 10.

(b) *Maple sugar, maple cream, etc.*—Proceed as directed under 35.

55

POLARIZATION.—TENTATIVE.

(a) *Direct at 20°C.*—Proceed as directed under VIII, 14.

(b) *Invert at 20°C.*—Proceed as directed under VIII, 14.

(c) *Invert at 87°C.*—Proceed as directed under 25 to detect commercial glucose.

56 REDUCING SUGARS AS INVERT SUGAR.—TENTATIVE.

(a) *Before inversion.*—Proceed as directed under VIII, 25, using an aliquot of the solution used for direct polarization, 55 (a), and only neutral lead acetate for clarification.

(b) *After inversion.*—Proceed as directed under VIII, 25, using an aliquot of the solution used for the invert polarization, 55 (b), and only neutral lead acetate for clarification.

SUCROSE.**57 By Polarization.—Tentative.**

Proceed as directed under VIII, 14 or 16.

58 By Reducing Sugars Before and After Inversion.—Tentative.

Proceed as directed under VIII, 18.

59 TOTAL ASH.—TENTATIVE.

Proceed as directed under 13.

60 SOLUBLE AND INSOLUBLE ASH.—TENTATIVE.

Proceed as directed under 17.

61 ALKALINITY OF THE SOLUBLE ASH.—TENTATIVE.

Proceed as directed under 18.

62 ALKALINITY OF THE INSOLUBLE ASH.—TENTATIVE.

Proceed as directed under 19.

LEAD NUMBER (WINTON).—TENTATIVE.**63 REAGENTS.**

Standard basic lead acetate solution.—Boil 430 grams of normal lead acetate and 130 grams of litharge, for 30 minutes, or boil 560 grams of Horne's dry basic lead acetate with 1 liter of water, cool, allow to settle and dilute the supernatant liquid to 1.25 sp. gr. To a measured amount of this solution add 4 volumes of water and filter if not perfectly clear. The solution should be standardized each time a set of determinations is made.

If the directions for preparing the basic lead acetate are not carried out carefully, the use of Horne's dry basic lead acetate is preferable.

64 DETERMINATION OF LEAD IN THE BLANK.

Transfer 25 cc. of the standard basic lead acetate to a 100 cc. flask, add a few drops of acetic acid, and make up to the mark with water. Shake and determine lead sulphate in 10 cc. of the solution as directed under 65. The use of the acid is imperative in this case to keep the lead in solution, when diluted with water.

65 DETERMINATION.

Transfer 25 grams of the sample to a 100 cc. flask by means of water. Add 25 cc. of the standard basic lead acetate and shake, fill to the mark, shake, and allow to stand for at least 3 hours before filtering. Pipette 10 cc. of the clear filtrate into a 250 cc. beaker, add 40 cc. of water and 1 cc. of concentrated sulphuric acid, shake and add 100 cc. of 95% alcohol. Allow to stand overnight, filter on a tared Gooch,

wash with 95% alcohol, dry in a water oven, and ignite in a muffle or over a Bunsen burner, applying the heat gradually at first, and avoiding a reducing flame. Cool and weigh. Subtract the weight of lead sulphate so found from the weight of lead sulphate found in the blank, 64, and multiply by the factor 27.325. The use of this factor gives the lead number directly without the various calculations otherwise required.

MALIC-ACID VALUE.

66

Cowles Method.¹⁴—Tentative.

Weigh 6.7 grams of the sample into a 200 cc. beaker, add 5 cc. of water, then 2 cc. of a 10% calcium acetate solution and stir. Add gradually, and with constant stirring, 100 cc. of 95% alcohol, and agitate the solution until the precipitate settles, or let stand, until the supernatant liquid is clear. Filter off the precipitate and wash with 75 cc. of 85% alcohol. Dry the filter paper and ignite in a platinum dish. Add 10 cc. of N/10 hydrochloric acid and warm gently until all the lime dissolves. Cool and titrate back with N/10 sodium hydroxid, using methyl orange as an indicator. The difference in cc. divided by 10 represents the malic acid value of the sample. Previous to use the reagents should be tested by a blank determination and any necessary corrections applied.

67

METALS.—TENTATIVE.

Proceed as directed under XII.

SUGAR HOUSE PRODUCTS.

SUCROSE IN BEETS.

68

Alcohol Extraction Method (Hersfeld Modification¹⁵).—Tentative.

Weigh 26 grams of the beet pulp and transfer to a 100 cc. flask with about 50 cc. of 90% alcohol and 3–5 cc. of basic lead acetate solution. Connect a reflux condenser to the flask and place on a boiling water bath for 10–15 minutes. Then pour the whole into a Soxhlet extractor, washing out the flask with fresh portions of 90% alcohol. Connect the same 100 cc. flask to the extractor, and fit the latter with a return condenser. Add 90% alcohol until the siphon is started and the flask is about three fourths full. Place the flask in a covered water bath kept at a heat that will allow the alcohol to boil freely. Continue the extraction for 1–4 hours, or until a test of the alcohol in the extractor gives no color with alpha-naphthol solution when tested as follows: Introduce into a test tube a few drops of the alcohol coming from the extractor, add 4 or 5 drops of a 20% alcoholic alpha-naphthol solution and 2 cc. of water. Shake well, tip the tube, and allow 2–5 cc. of colorless concentrated sulphuric acid to flow down the side of the tube; then hold the tube upright and, if sucrose is present, a color varying from a faint to a deep violet will be noted at the junction of the two liquids. On shaking, the whole solution becomes a blue violet color. This test is suitable for this work, but it must be remembered that other substances besides sucrose give this color reaction.

Remove the flask, transfer to a 100 cc. graduated flask, cool to the standard temperature, dilute to the mark with 90% alcohol, shake and filter, keeping the funnel covered with a watch glass. Polarize in a 200 mm. tube.

Avoid evaporation and changes of temperature and also use a minimum amount of basic acetate for clarification, 3 cc. rather than 5 cc. By digesting the beet pulp with the alcohol before extraction, the time of extraction is greatly shortened the pulp becomes thoroughly impregnated with the alcohol, and all the air is removed, resulting in a good extraction of the whole material. If the pulp is fine

and tends to clog the siphon, alcohol-washed cotton may be used as a plug in the extractor before adding the beet pulp, and a fine mesh screen may be placed over the pulp to keep the whole compact in the extractor.

69

*Pellet Aqueous Method*¹⁶ (*Hot Digestion*).—*Tentative.*

Weigh 52 grams of the beet cuttings and transfer them with water to a wide-mouthed flask graduated to a content of 201.2 cc.; add 5–10 cc. of basic lead acetate solution, fill the flask to the mark with hot water, and shake. Immerse the flask in a water bath at 80°C. and rotate at intervals. Add water from time to time so that at the end of the heating (about 30 minutes) the water in the flask is a little above the mark. Remove the flask from the water bath and allow it to cool to standard temperature. Add sufficient concentrated acetic acid to make the solution very slightly acid (generally less than 0.5 cc.) and a few drops of ether to break the foam. Make up to the mark, mix thoroughly, filter, and polarize in a 200 mm. tube.

The fineness of the pulp governs the time of heating. Add enough water at the start and maintain this volume during the extraction, so that not more than 5 cc. of water will be necessary to complete the volume after cooling. The proportion of pulp to water must not be increased beyond the prescribed amount, for when smaller proportions of water to pulp are used and then a large quantity of water is added at the last to make up to volume, the sugar does not become equally diffused and the results are too low. Differences of over 1% in sugar content may be caused by lack of care in this particular.

70

Hot Water Digestion Method.—*Tentative.*

(*Hersfeld Modification of the Sachs Le Docte Method*¹⁷.)

There are needed nickel-plated sheet iron vessels, 11 cm. high, 6 cm. body diameter, and 4 cm. mouth diameter, also stoppers covered with tin foil to fit the same.

Weigh 26 grams of the beet pulp on a watch glass (small enough to go into the neck of the beaker) and transfer to the metal beaker, add 177 cc. of dilute basic lead acetate solution (5 parts of basic lead acetate solution (sp. gr. 1.25) to 100 parts of water), shake and stopper lightly. Submerge the beaker in a water bath at 75°–80°C. for 30 minutes, shaking intermittently. When all the air has been expelled (generally after 5 minutes), tighten the stopper. After 30 minutes, shake, cool to standard temperature, filter, add a drop of acetic acid to the filtrate and polarize in a 400 mm. tube. The reading is the per cent of sugar in the beet pulp.

BIBLIOGRAPHY.

- ¹ Browne. Handbook of Sugar Analysis. 1912, p. 16.
- ² Wiss. Abh. der Kaiserlichen Normal-Eichungs-Kommission, 1900, 2: 153; U. S. Bur. Standards, Circ. 19, 5th ed., p. 26.
- ³ Intern. Sugar J., 10: 69; U. S. Bur. Chem. Bull. 122, p. 169.
- ⁴ Z. anal. Chem., 1899, 38: 345.
- ⁵ Leach. Food Inspection and Analysis. 1913, p. 624.
- ⁶ Analyst, 1896, 21: 182.
- ⁷ Leach. Food Inspection and Analysis. 1913, p. 622.
- ⁸ U. S. Bur. Chem. Bulls. 110 and 154; Z. Nahr. Genussm., 1909, 18: 625.
- ⁹ U. S. Bur. Chem. Bull. 110, p. 60.
- ¹⁰ Ibid., 110 and 154.
- ¹¹ Ibid., 154, p. 15.
- ¹² Analyst, 1911, 36: 586.
- ¹³ Z. Nahr. Genussm., 1910, 19: 72.
- ¹⁴ J. Am. Chem. Soc., 1908, 30: 1285.
- ¹⁵ U. S. Bur. Chem. Bull. 146, p. 17.
- ¹⁶ Ibid., p. 18.
- ¹⁷ Ibid., p. 19.

X. FOOD PRESERVATIVES.—TENTATIVE.

SALICYLIC ACID.

1

PREPARATION OF SAMPLE.

(a) *Non-alcoholic liquids*.—Many liquids may be extracted directly as described in 2 or 4 without further treatment. If gums or mucilaginous substances are present, pipette 100 cc. into a 250 cc. volumetric flask, add about 5 grams of sodium chlorid, shake until the latter is dissolved, make up to the mark with alcohol, shake vigorously, allow the mixture to stand for 10 minutes with occasional shaking, filter through a dry folded filter and treat an aliquot of the filtrate as directed under (b).

(b) *Alcoholic liquids*.—Make 200 cc. of the sample alkaline with sodium hydroxid solution, using litmus as an indicator, and evaporate on a steam bath to about one third its original volume. Dilute to the original volume with water and filter, if necessary, through a dry filter.

(c) *Solid or semi-solid substances*.—Grind the sample and mix thoroughly. Transfer a convenient quantity (50–200 grams according to the consistency of the sample) to a 500 cc. volumetric flask, add sufficient water to make a volume of about 400 cc., shake until the mixture becomes uniform, add 2–5 grams of calcium chlorid, shake until the latter is dissolved, render distinctly alkaline with sodium hydroxid solution, using litmus as an indicator, fill to the mark with water, shake thoroughly, allow to stand for at least 2 hours shaking frequently and filter through a large folded filter.

DETECTION AND ESTIMATION.

2

Ferric Chlorid Test.—Qualitative.

Introduce 50 cc. of the sample or an equivalent amount of an aqueous extract, prepared as directed under 1, into a separatory funnel, add one tenth its volume of dilute hydrochloric acid (1 to 3) and extract with 50 cc. of ether. If the mixture emulsifies, add 10–15 cc. of petroleum ether (b. p. below 60°C.) and shake. If this treatment fails to break the emulsion whirl the mixture in a centrifuge, or allow it to stand until a considerable portion of the aqueous layer has separated, run off the latter, shake vigorously and again allow to separate. Wash the ether layer with two 5 cc. portions of water, evaporate the greater portion of the ether in a porcelain dish on a steam bath, allow the remainder to evaporate spontaneously and add a drop of 0.5% ferric chlorid solution. A violet color indicates salicylic acid.

If coloring matter or other interfering substances are present in the residue left after evaporation of the ether, purify the salicylic acid by one of the following methods:

(a) Dissolve the residue from the ether extract, obtained as directed above, in about 25 cc. of ether, transfer the latter to a separatory funnel and shake with an equal quantity of water, made distinctly alkaline with several drops of ammonium hydroxid. Allow to separate, filter the aqueous layer through a wet filter into a porcelain dish, evaporate almost to dryness, and test the residue as directed above.

(b) Dry the residue from the ether extract, obtained as directed above, in a desiccator over sulphuric acid and extract with several 10 cc. portions of carbon

disulphid or petroleum ether (b. p. below 60°C.), rubbing the contents of the dish with a glass rod, and filtering the successive portions of the solvent through a dry paper into a second porcelain dish. Evaporate the greater portion of the solvent on a steam bath, allow the remainder to evaporate spontaneously and test the residue as directed above.

(C) Transfer the residue from the ether extract, obtained as directed above, to a small porcelain crucible by means of a few cc. of ether and allow the solvent to evaporate spontaneously. Cut a hole in a piece of asbestos board sufficiently large to admit about two thirds of the crucible, cover the latter with a small, round-bottomed flask filled with cold water, and heat over a small Bunsen flame until any salicylic acid present has sublimed and condensed upon the bottom of the flask. Test the sublimate as directed above.

3*Jorissen's Test.*—Qualitative.

Dissolve the residue from the ether extract, obtained as directed under 2, or, in case impurities are present, the purified material obtained as directed under 2 (a), (b) or (c) in a little hot water. Cool 10 cc. of the solution in a test tube, add 4 or 5 drops of 10% potassium nitrite solution, 4 or 5 drops of 50% acetic acid and 1 drop of 10% cupric sulphate solution, mix thoroughly and heat to boiling. Boil for half a minute and allow to stand for 1-2 minutes. In the presence of salicylic acid a blood red color will develop.

Colorimetric Method.—Quantitative.**4****EXTRACTION.**

Pipette a convenient portion of the sample (100 cc. or a volume representing not less than 20 grams of the original sample) or a solution, prepared as in 1, into a separatory funnel, make the solution neutral to litmus with dilute hydrochloric acid (1 to 3) and add an excess of concentrated hydrochloric acid equivalent to 2 cc. of acid for each 100 cc. of solution. Extract with 4 separate portions of ether, using for each extraction a volume of ether equivalent to half the volume of the aqueous layer. If an emulsion forms on shaking, this may usually be broken by adding a little (one fifth the volume of the ether layer) petroleum ether (b. p. below 60°C.) and shaking again or by centrifugalizing. If an emulsion still persists, allow it to remain with the aqueous layer. If an emulsion remains after the fourth extraction, separate it from the clear ether and the clear aqueous layer and extract it separately with 2-3 small portions of ether. Combine the ether extracts, wash with one tenth their volume of water, allow the layers to separate and reject the aqueous layer. Wash in this way until the aqueous layer after separation yields a yellow color upon the addition of methyl orange and 2 drops of N/10 sodium hydroxid. Distil slowly the greater part of the ether, transfer the remainder to a porcelain dish and allow the ether to evaporate spontaneously. If there are no interfering substances present, proceed as directed in 5. If such interfering substances are present, purify the residue by one of the following methods:

(a) Dry thoroughly the residue in vacuo over sulphuric acid and extract with 10 portions of 10-15 cc. each of carbon disulphid or petroleum ether (b. p. below 60°C.), rub the contents of the dish with a glass rod and filter the successive portions of the solvent through a dry filter into a porcelain dish. Test the extracted residue with a drop of ferric alum solution and, if it gives a reaction for salicylic acid, dissolve it in water and reextract with ether, proceeding as directed above. Distil the greater portion of the carbon disulphid or petroleum ether and allow the remainder to evaporate spontaneously. Proceed as directed in 5.

(b) Dissolve the residue in 40–50 cc. of ether. Transfer the ether solution to a separatory funnel and extract with 3 successive 15 cc. portions of 1% ammonium hydroxid. (If fat is known to be present in the original ether extract, extract the latter directly with 4 portions of the ammonium hydroxid instead of 3.) Combine the alkaline aqueous extracts, acidify, again extract with ether and wash the combined ether extracts as directed above. Distil slowly the greater portion of the ether, allow the remainder to evaporate spontaneously and proceed as directed in 5.

5

DETERMINATION.

Dissolve the residue, obtained in 4, in a small amount of hot water and, after cooling, dilute to a definite volume (usually 50–100 cc.), dependent on the amount of salicylic acid present. If the solution is not clear, filter through a dry filter. Dilute aliquots of the solution and treat with a few drops of 0.5% ferric chlorid solution or 2% ferric alum solution.

The ferric alum solution should be boiled until a precipitate appears, allowed to settle, and filtered. The acidity of the solution is slightly increased in this manner, but it remains clear for a considerable time, and the turbidity caused by its dilution with water is much less and does not appear as soon as when the unboiled solution is used. This turbidity interferes with the exact matching of the color.

Compare the colors developed with that obtained when a standard salicylic acid solution (containing 1 mg. of salicylic acid in 50 cc.) is similarly treated, using Nessler tubes or a colorimeter. In either case, and especially with ferric chlorid, avoid an excess of the reagent, although an excess of 0.5 cc. of 2% ferric alum solution may be added to 50 cc. of the comparison solution of salicylic acid without impairing the results.

BENZOIC ACID.

PREPARATION OF SAMPLE.

6

General Method.

If solid or semi-solid, grind the sample, and mix thoroughly. Transfer about 150 grams to a 500 cc. graduated flask, add enough pulverized sodium chlorid to saturate the water in the sample, render alkaline with sodium hydroxid solution or milk of lime, and dilute to the mark with a saturated salt solution. Allow to stand for at least 2 hours, with frequent shaking, and filter. If the sample contains large amounts of matter precipitable by salt solution, it is advisable to follow a method similar to that given under 7 (d). When alcohol is present, follow the method given under 7 (c). When large amounts of fats are present, make an alkaline extraction of the filtrate before proceeding as directed under 11.

7

Special Methods.

(a) *Ketchup*.—Saturate the water in 150 grams of ketchup by adding 15 grams of pulverized sodium chlorid. Transfer the mixture to a 500 cc. graduated flask, rinsing with about 150 cc. of saturated sodium chlorid solution. Make slightly alkaline to litmus paper with strong sodium hydroxid solution and fill to the mark with saturated salt solution. Allow to stand for at least 2 hours, shaking frequently. Squeeze through a heavy muslin bag and then filter through a large folded filter.

(b) *Jellies, jams, preserves and marmalades*.—Dissolve 150 grams of the sample in about 300 cc. of saturated salt solution. Add 15 grams of pulverized sodium chlorid. Make alkaline to litmus paper with milk of lime. Transfer to a 500 cc. graduated flask and dilute to the mark with saturated salt solution. Allow to

stand for at least 2 hours, shaking frequently, centrifugalize if necessary, and filter through a large folded filter.

(C) *Cider containing alcohol, and similar products.*—Make 250 cc. of the sample alkaline to litmus paper with sodium hydroxid solution and evaporate on the steam bath to about 100 cc. Transfer the sample to a 250 cc. graduated flask, add 30 grams of pulverized sodium chlorid and shake until dissolved. Dilute to the original volume, 250 cc., with saturated salt solution, allow to stand for at least 2 hours, shaking frequently, and filter through a folded filter.

(d) *Salted or dried fish.*—Wash 50 grams of the ground sample into a 500 cc. graduated flask with water. Make slightly alkaline to litmus paper with strong sodium hydroxid solution and dilute to the mark with water. Allow to stand for at least 2 hours, shaking frequently, and then filter through a folded filter. Pipette accurately as large a portion of the filtrate as possible (at least 300 cc.) into a second 500 cc. flask. Add 30 grams of the pulverized sodium chlorid for each 100 cc. of solution. Shake until the salt has dissolved and dilute to the mark with saturated salt solution. Mix thoroughly and filter off the precipitated protein matter on a folded filter.

8

DETECTION AND ESTIMATION.

Extract benzoic acid as directed under 2 or 4. If benzoic acid is present in considerable quantity, it will crystallize from the ether in shining leaflets having a characteristic odor on heating. Dissolve the residue in hot water, divide into 2 portions, and test according to 9 or 10.

9

Ferric Chlorid Test.—Qualitative.

Make the solution from 8 alkaline with ammonium hydroxid, expel the excess of ammonia by evaporation, dissolve the residue in water, and add a few drops of a neutral 0.5% ferric chlorid solution. A brownish precipitate of ferric benzoate indicates the presence of benzoic acid.

10

Modified Mohler Test.—Qualitative.

Add to the water solution, prepared as described under 8, 1-3 cc. of N/3 sodium hydroxid and evaporate to dryness. To the residue, add 5-10 drops of concentrated sulphuric acid and a small crystal of potassium nitrate. Heat for 10 minutes in a glycerol bath at 120°-130°C., or for 20 minutes in a boiling water bath. The temperature must not exceed 130°C. After cooling add 1 cc. of water and make distinctly ammoniacal; boil the solution to decompose any ammonium nitrite which may have been formed. Cool and add a drop of fresh, colorless ammonium sulphid, without allowing the layers to mix. A red-brown ring indicates benzoic acid. On mixing, the color diffuses through the whole liquid and, on heating, finally changes to greenish yellow. This differentiates benzoic acid from salicylic acid or cinnamic acid. The last two form colored compounds, which are not destroyed by heating. The presence of phenolphthalein interferes with this test.

11

Quantitative Method.

Pipette a convenient portion (100-200 cc.) of the filtrate, obtained in 6 or 7, into a separatory funnel. Neutralize the solution to litmus paper with hydrochloric acid (1 to 3) and add an excess of 5 cc. of the same acid. In the case of salted fish a precipitation of protein matter usually occurs on acidifying, but the precipitate does not interfere with the extraction. Extract carefully with chloroform, using successive portions of 70, 50, 40, and 30 cc. To avoid an emulsion, shake cautiously

each time. The chloroform layer usually separates readily after standing a few minutes. If an emulsion forms, break it: (1) by stirring the chloroform layer with a glass rod; (2) by drawing it off into a second funnel and giving 1 or 2 sharp shakes from one end of the funnel to the other; or (3) by centrifugalizing for a few moments. As this is a progressive extraction, draw off carefully as much of the clear chloroform solution as possible after each extraction, but do not draw off any of the emulsion with the chloroform layer. If this precaution is taken, the chloroform extract need not be washed.

Transfer the combined chloroform extracts to a porcelain evaporating dish, rinse the container several times with a few cc. of chloroform, and evaporate to dryness at room temperature in a current of air dried over calcium chlorid.

The extract may also be transferred from the separatory funnel to a 300 cc. Erlenmeyer flask, rinsing the separatory funnel 3 times with 5-10 cc. of chloroform. Distil very carefully to about one fourth the original volume, keeping the temperature down so that the chloroform comes over in drops, not in a steady stream. Then transfer the residue to a porcelain evaporating dish, rinsing the flask 3 times with 5-10 cc. portions of chloroform, and allow to evaporate to dryness spontaneously.

Dry the residue overnight (or until no odor of acetic acid can be detected if the product is a ketchup) in a desiccator containing sulphuric acid. Dissolve the residue of benzoic acid in 30-50 cc. of neutral alcohol, add about one fourth this volume of water, 1 or 2 drops of phenolphthalein, and titrate with N/20 sodium hydroxid (1 cc. is equivalent to 0.0072 gram of anhydrous sodium benzoate).

SACCHARIN.

12

Qualitative Test.

Extract with ether (after maceration and exhaustion with water, if necessary), as directed in 1 and 4. Allow the ether extract to evaporate spontaneously and note the taste of the residue. The presence of saccharin, to the extent of 20 mg. per liter, is indicated by a sweet taste. Confirm by heating with sodium hydroxid, as described below, and detecting the salicylic acid formed thereby. A sweet taste, suggesting the presence of a trace of saccharin, has been obtained frequently in saccharin-free wines, due to the so-called "false saccharin".

Acidify 50 cc. of a liquid food or the aqueous extract of 50 grams of a solid or semi-solid, prepared as directed in 1 (C), and extract with ether as directed in 13. Dissolve the residue, remaining after evaporation of the ether, in a little hot water and test a small portion of this solution for salicylic acid as directed under 2 or 3. Dilute the remainder of the solution to about 10 cc., and add 2 cc. of sulphuric acid (1 to 3). Heat to boiling and add a slight excess of 5% potassium permanganate solution, drop by drop; partly cool the solution, dissolve a piece of sodium hydroxid in it, and filter the mixture into a silver dish (silver crucible lids are well adapted to the purpose); evaporate to dryness and heat for 20 minutes at 210°-215°C. Dissolve the residue in water, acidify with hydrochloric acid and test the ether extract for salicylic acid as directed under 2 or 3. By this method all the so-called "false saccharin" and the salicylic acid naturally present (also added salicylic acid when not present in too large an amount) are destroyed, while 5 mg. of saccharin per liter are detected with certainty.

13

Quantitative Method.

Pipette 100 cc. of the sample, or a convenient portion of a solution, prepared as directed under 1, representing not less than 20 grams of the sample, into a sepa-

ratory funnel, make the solution neutral to litmus with dilute hydrochloric acid (1 to 3) and then add concentrated hydrochloric acid in the proportion of 5 cc. for each 100 cc. of solution. Extract with 4 separate portions of ether using, for each extraction, a volume of ether equivalent to half the volume of the aqueous layer. If the mixture emulsifies on shaking, this difficulty may be overcome as directed under 4. Wash the combined ether extracts with two 5 cc. portions of water, remove the ether by distillation, and transfer the residue into a platinum crucible by means of a small amount of ether. Evaporate the ether on a steam bath, add about 2-3 cc. of 10% sodium carbonate solution to the residue, rotate so that all of the residue is brought into contact with the solution, and evaporate to dryness on a steam bath. Add 4 grams of a mixture of equal parts of anhydrous sodium and potassium carbonates, heat gently at first, and then to complete fusion for 30 minutes over an alcohol or other sulphur-free flame. Cool, dissolve the melt in water, acidify with hydrochloric acid and determine the sulphate present as barium sulphate. Correct the result thus obtained for any sulphur present in the fusion mixture as found in a blank determination. Calculate the amount of saccharin in the sample by multiplying the weight of barium sulphate by 0.7845.

BORIC ACID AND BORATES.

14

*Qualitative Test.**

Preliminary test.—Immerse a strip of turmeric paper in the sample acidified with hydrochloric acid in the proportion of 7 cc. of concentrated acid to each 100 cc. of sample, and allow the paper to dry spontaneously. If borax or boric acid is present, the paper will acquire a peculiar red color, changed by ammonium hydroxid to a dark blue-green but restored by acid. Solid or pasty samples may be heated with enough water to make them sufficiently fluid, concentrated hydrochloric acid added in about the proportion of 1 to 13 and the liquid tested in the same way.

Confirmatory test.—Make about 25 grams of the sample decidedly alkaline with lime water and evaporate to dryness on a water bath. Ignite the residue to destroy organic matter. Digest with about 15 cc. of water, add concentrated hydrochloric acid, drop by drop, until the ignited residue is dissolved, and then add 1 cc. in excess. Saturate a piece of turmeric paper with the solution, and allow it to dry without the aid of heat. In the presence of borax or boric acid, the color change will be the same as given above.

15

Quantitative Method.†

Make 10-100 grams of the sample (depending upon the nature of the sample and the amount of boric acid present) distinctly alkaline with sodium hydroxid solution and evaporate to dryness in a platinum dish. Ignite the residue until organic matter is destroyed, avoiding an intense red heat, cool, digest with about 20 cc. of hot water, and add hydrochloric acid, drop by drop, until the reaction is distinctly acid. Filter into a 100 cc. flask, and wash with a little hot water, the volume of the filtrate not to exceed 50-60 cc. Return the filter containing any unburned carbon to the platinum dish, make alkaline by wetting thoroughly with lime water, dry on a steam bath and ignite to a white ash. Dissolve the ash in a few cc. of dilute hydrochloric acid and add to the liquid in the 100 cc. flask, rinsing the dish with a few cc. of water. To the combined solutions, add 0.5 gram of calcium chlorid and a few drops of phenolphthalein, then 10% sodium hydroxid solution until a permanent light pink color is produced, and finally dilute to

the mark with lime water. Mix and filter through a dry filter. To 50 cc. of the filtrate add N/1 sulphuric acid until the pink color disappears, then add methyl orange, and continue the addition of the acid until the yellow color is changed to pink. Boil for about 1 minute to expel carbon dioxid. Cool, and carefully add N/5 sodium hydroxid until the liquid assumes a yellow tinge, avoiding an excess of the alkali. All the boric acid is now in a free state with no uncombined sulphuric acid present. Add a little phenolphthalein, and an equal volume of neutral glycerol. Titrate with N/5 sodium hydroxid until a permanent pink color is produced. About 10 grams of mannitol may be substituted for the glycerol in this determination. At the end of the titration add an additional 2 grams and continue the titration if the pink color is discharged. Repeat the alternate addition of mannitol and alkali until a permanent end point is reached.

One cc. of N/5 sodium hydroxid is equivalent to 0.0124 gram of boric acid.

FORMALDEHYDE.

16

PREPARATION OF SAMPLE.

If solid or semi-solid, macerate 200-300 grams of the material with about 100 cc. of water in a mortar. Transfer to a short-necked, 500-800 cc. copper or glass distillation flask and make distinctly acid with phosphoric acid, connect with a condenser and distil 40-50 cc. In the case of highly colored liquids, the same method of preparation should be employed.

In the case of meats and fats, extract the formaldehyde with alcohol and use the filtrate. In the case of fat, heat the mixture above the melting point of the fat to insure thorough extraction. In the case of milk, shake with an equal volume of strong alcohol and use the filtrate. Shake other liquids with an equal volume of strong alcohol and filter from any insoluble matter.

QUALITATIVE TESTS.

17

Phenylhydrazin Hydrochlorid Method.⁵

Mix 5 cc. of the distillate, as prepared under 16, or of an alcoholic solution or extract obtained as directed above, with 0.03 gram of phenylhydrazin hydrochlorid, and 4 or 5 drops of a 1% ferric chlorid solution. Add slowly and with agitation, in a bath of cold water to prevent heating the liquid, 1-2 cc. of concentrated sulphuric acid. Dissolve the precipitate by the addition either of concentrated sulphuric acid (keeping the mixture cool) or alcohol. In the presence of formaldehyde a red color develops.

This method gives reliable reactions for formaldehyde in solutions of formaldehyde varying from 1 part in 50,000 to 1 part in 150,000. Acetaldehyde and benzaldehyde give no reaction when treated by this method and do not interfere with the reaction given by formaldehyde.

18

Hehner Method.⁶

Mix about 5 cc. of the distillate, obtained in 16, with an equal volume of pure milk, or a 1-2% solution of egg albumen, in a test tube and underlay with strong commercial sulphuric acid without mixing. A violet or blue color at the junction of the two liquids indicates formaldehyde. This color is given only in the presence of a trace of ferric chlorid or other oxidizing agent. As pointed out by Hehner, milk may be treated directly by this method and gives positive tests in the presence of 1 or more parts of formaldehyde per 10,000. Some other articles of food rich in proteins, for example, egg albumen, give the reaction in the presence of water without the addition of milk.

19

Leach Method.

Mix about 5 cc. of the distillate, obtained under **16**, with an equal volume of pure milk in a porcelain casserole and add about 10 cc. of concentrated hydrochloric acid, containing 1 cc. of 10% ferric chlorid solution, to each 500 cc. of acid. Heat to 80°-90°C. directly over the gas flame, rotating the casserole to break up the curd. A violet coloration indicates formaldehyde.

Rimini Method.¹

20

Phenylhydrazin Hydrochlorid and Sodium Nitro-prussid Test.

This method may be applied directly to liquid foods, to an aqueous or alcoholic extract of solid foods, or to the distillate prepared as directed in **16**. In the case of milk, apply the method directly. In the case of meat, comminute the sample, extract with 2 volumes of hot water, and employ the expressed liquid for the test. Heat fats above their melting point with 10 cc. of alcohol, shake thoroughly, cool, filter through a moistened filter, and use the filtrate for the test.

Dissolve a lump of phenylhydrazin hydrochlorid about the size of a pea in 3-5 cc. of the liquid to be tested, add 2-4 drops (not more) of a 5-10% sodium nitro-prussid solution and 8-12 drops of an approximately 12% sodium hydroxid solution. If formaldehyde is present, a green or blue color develops depending upon the amount. When formaldehyde is present to the extent of more than 1 part in 70,000-80,000 in the solution tested, a distinct green or bluish green reaction is obtained. In more dilute solutions the green tint becomes less marked and a yellow tinge tending toward greenish brown develops.

With this method acetaldehyde and benzaldehyde give a color varying from red to brown, according to the strength of the solution. A reaction may therefore be obtained with these aldehydes similar to that obtained with formaldehyde in solutions more dilute than 1 part in 70,000. The presence of acetaldehyde or benzaldehyde together with formaldehyde gives a yellowish or yellowish green tinge. The reaction for formaldehyde may therefore be masked by the presence of other aldehydes, but is characteristic when a clear green color is obtained.

21

Phenylhydrazin Hydrochlorid and Potassium Ferricyanid Test.

Proceed as directed in **20**, substituting a solution of potassium ferricyanid for the sodium nitro-prussid. Formaldehyde gives a red color. Alcoholic extracts from foods must be diluted with water to prevent the precipitation of potassium ferricyanid. The test is not applicable in the presence of the coloring matter of blood.

22

Phenylhydrazin Hydrochlorid and Ferric Chlorid Test.

Treat 15 cc. of milk or other liquid food or of the distillate, prepared as directed under **16**, with 1 cc. of a dilute phenylhydrazin hydrochlorid solution, then with a few drops of dilute ferric chlorid solution and, finally, with concentrated hydrochloric acid. The presence of formaldehyde is indicated by the formation of a red color, which changes after some time to orange yellow.

Milk may be examined directly by this method, but more delicate tests may be obtained from the distillate from milk or from milk serum. Acetaldehyde or benzaldehyde does not interfere with the reaction.

23

*Phloroglucol Method.*⁴

To 10 cc. of milk or other liquid food under examination in a test tube add, by means of a pipette, 2 cc. of phloroglucol reagent (1 gram of phloroglucol, 20 grams of sodium hydroxid and water to make 100 cc.), placing the end of the pipette on the bottom of the tube in such a manner that the reagent will form a separate layer.

If formaldehyde be present, a bright red coloration (not purple) forms at the zone of contact. This solution gives a yellow color in the presence of some aldehydes, and, if it is used for the detection of aldehyde formed by the oxidation of methyl alcohol after the destruction of ethyl aldehyde with hydrogen peroxid, an orange yellow color will slowly appear when an insufficient amount of hydrogen peroxid has been employed. On the other hand, if the excess of hydrogen peroxid is not fully destroyed before the use of this reagent, a purple color develops slowly. The clear, red color given by the use of this reagent forms quickly, and, in the presence of but a small amount of formaldehyde fades rapidly.

FLUORIDS.**QUALITATIVE TESTS.**

24

*Method I.—Modified Method of Blarez.*⁹

Thoroughly mix the sample and boil 150 cc. (in the case of solid foods an aqueous extract may be employed provided the fluorids are in a soluble form). Add to the boiling liquid 5 cc. of 10% potassium sulphate solution and 10 cc. of 10% barium acetate solution. Collect the precipitate in a compact mass (a centrifuge may be used advantageously) and wash upon a small filter. Transfer to a platinum crucible and ignite.

Dip a carefully cleaned glass plate, while hot, in a mixture of equal parts of Carnuba wax and paraffin and allow to cool. Make, with a sharp instrument, a distinctive mark through the wax, taking care not to scratch the surface of the glass.

Add a few drops of concentrated sulphuric acid to the residue in the crucible and cover with the waxed plate, having the mark nearly over the center and making sure that the edge of the crucible is in close contact with it. Keep the top surface of the plate cool by means of a suitable device and heat the crucible for an hour at as high a temperature as practicable without melting the wax (an electric stove gives the most satisfactory form of heat).

If fluorids be present, a distinct etching will be apparent on the glass where it was exposed.

25

Method II.

The preceding method may be varied by mixing a small amount of precipitated silica with the precipitated barium fluorid and applying the method for the detection of fluosilicates, under 27 or 28.

This method is of value in the case of foods whose ash contains a considerable amount of silica. Under these circumstances, concentrated sulphuric acid liberates silicon fluorid, which would escape detection under 24.

FLUOBORATES AND FLUOSILICATES.

26

PREPARATION OF SAMPLE.

Make about 200 grams of the sample alkaline with lime water, evaporate to dryness, and incinerate. Extract the crude ash with water, to which sufficient acetic

acid has been added to decompose carbonates, filter, ignite the insoluble portion, extract with dilute acetic acid, and again filter. The insoluble portion now contains calcium silicate and fluorid, while the filtrate will contain all the boric acid present.

QUALITATIVE TESTS.

27

Method I.¹⁰

Incinerate the filter, from 26, containing the insoluble portion, mix with a little precipitated silica, transfer to a short test tube, attached to a small U-tube containing a few drops of water and add 1-2 cc. of concentrated sulphuric acid. Keep the test tube in a beaker of water on the steam bath for 30-40 minutes. If any fluorin be present, the silicon fluorid generated will be decomposed by the water in the U-tube and will form a gelatinous deposit on the walls of the tube.

Next test the filtrate as directed under 14. If both hydrofluoric and boric acids be present, it is probable that they are combined as borofluorid. If, however, silicon fluorid is detected and not boric acid, the operation should be repeated without the introduction of the silica, in which case the formation of the silicon skeleton is conclusive evidence of the presence of fluosilicate. In an ash containing an appreciable amount of silica, sulphuric acid will liberate silicon fluorid rather than hydrofluoric acid. The presence of a fluosilicate is indicated, therefore, and not the presence of a fluorid.

28

Method II.

Incinerate the filter, from 26, containing the insoluble portion, in a platinum crucible, mix with a little precipitated silica, and add 1 cc. of concentrated sulphuric acid. Cover the crucible with a watch glass, from the underside of which a drop of water is suspended, and heat for an hour at 70°-80°C., keeping the watch glass cooled. The silicon fluorid which is formed is decomposed by the water, leaving a gelatinous deposit of silica and etching a ring at the periphery of the drop of water. Test the filtrate for boric acid as directed under 14.

SULPHUROUS ACID.

29

Qualitative Test.¹¹

Add some sulphur-free zinc, and several cc. of hydrochloric acid to about 25 grams of the sample (with the addition of water, if necessary) in a 200 cc. Erlenmeyer flask. In the presence of sulphites, hydrogen sulphid will be generated and may be detected with lead acetate paper. Traces of metallic sulphids are occasionally present in vegetables, and will give the same reaction as sulphites under the conditions of the above test. Positive results obtained by this method should be verified by the distillation method under 30.

It is always advisable to make the quantitative determination of sulphites, owing to the danger that the test may be due to traces of sulphids. A trace is not to be considered sufficient indication of the presence of sulphur dioxid either as a bleaching agent or as a preservative.

TOTAL SULPHUROUS ACID.

30

Method I.—Distillation Method.

Distil 20-100 grams of the sample (adding recently boiled water if necessary) in a current of carbon dioxid, after the addition of about 5 cc. of a 20% glacial phos-

phoric acid solution, until 150 cc. have passed over. Collect the distillate in about 100 cc. of nearly saturated bromin water, allowing the end of the condenser to dip below the surface. The method and apparatus may be simplified without material loss in accuracy by omitting the current of carbon dioxide, adding 10 cc. of phosphoric acid instead of 5 cc., and dropping into the distillation flask, immediately before attaching the condenser, a piece of sodium bicarbonate weighing not more than 1 gram. The carbon dioxide liberated is not sufficient to expel the air entirely from the apparatus, but will prevent oxidation to a large extent. When the distillation is finished, boil off the excess of bromin, dilute the solution to about 250 cc., add 5 cc. of hydrochloric acid (1 to 3), heat to boiling, and precipitate the sulphuric acid with 10% barium chlorid solution. Boil for a few minutes longer, allow to stand overnight in a warm place, filter on a weighed Gooch, wash with hot water, ignite at a dull red heat, and weigh as barium sulphate.

31

Method II.—Direct Titration Method.

In the examination of wine, fairly accurate results may be obtained by the following method:

Place 25 cc. of 5.6% potassium hydroxid solution in a 200 cc. flask. Introduce 50 cc. of the sample, mix with the potassium hydroxid solution, and allow the mixture to stand for 15 minutes with occasional agitation. Add 10 cc. of sulphuric acid (1 to 3) and a few cc. of starch solution, and titrate the mixture with N/50 iodine solution. Introduce the iodine solution as rapidly as possible and continue the addition until the blue color persists for several minutes. One cc. of N/50 iodine is equivalent to 0.00064 gram of sulphur dioxide.

DETERMINATION OF FREE SULPHUROUS ACID.

32

(Especially Adapted to Wine.)

Treat 50 cc. of the sample in a 200 cc. flask with about 5 cc. of sulphuric acid (1 to 3) add about 0.5 gram of sodium carbonate to expel the air, and titrate the sulphurous acid with N/50 iodine, as directed under 31.

BETA-NAPHTHOL.

33

Qualitative Test.

Extract 200 cc. of the sample, or of its aqueous extract, prepared as directed under 1 (C), with 10 cc. of chloroform in a separatory funnel. To the chloroform extract in a test tube add a few drops of alcoholic potash, and place in a boiling water bath for 2 minutes. The presence of beta-naphthol is indicated by the formation of a deep blue color, which changes to green and then to yellow.

ABRASTOL.

QUALITATIVE TESTS.

34

Sinibaldi Method.¹³

Make 50 cc. of the sample alkaline with a few drops of ammonium hydroxid and extract with 10 cc. of amyl alcohol, adding ethyl alcohol if an emulsion is formed. Decant the amyl alcohol, filter if turbid, and evaporate to dryness. Add to the residue 2 cc. of nitric acid (1 to 1), heat on the water bath until half of the liquid is evaporated, and transfer to a test tube with the addition of 1 cc. of water. Add about 0.2 gram of ferrous sulphate and an excess of ammonium hydroxid, drop by

drop, with constant shaking. If the resultant precipitate is of a reddish color, dissolve it in a few drops of sulphuric acid, and add ferrous sulphate and ammonium hydroxid as before. As soon as a dark colored or greenish precipitate is obtained, introduce 5 cc. of alcohol, dissolve the precipitate in sulphuric acid, shake well and filter. In the absence of abrastol this method gives a colorless or light yellow liquid, while a red color is produced in the presence of 0.01 gram of abrastol.

35

*Sanglé-Ferrière Method.*¹³

Boil 200 cc. of the sample with 8 cc. of concentrated hydrochloric acid for an hour in a flask fitted with a reflux condenser. Abrastol is thus converted into betanaphthol and is detected as directed under 33.

SUCROL OR DULCIN.

QUALITATIVE TESTS.

36

*Morpurgo Method.*¹⁴

Evaporate about 100 cc. of the sample, or of the aqueous extract prepared as directed under 1 (C) and neutralized with acetic acid, to a sirupy consistency after the addition of about 5 grams of lead carbonate, and extract the residue several times with 90% alcohol. Evaporate the alcoholic extract to dryness, extract the residue with ether, and allow the ether to evaporate spontaneously in a porcelain dish. Add 2 or 3 drops each of phenol and concentrated sulphuric acid and heat for about 5 minutes on the water bath, cool, transfer to a test tube and overlay with ammonium hydroxid or sodium hydroxid solution with the least possible mixing. The presence of dulcin is indicated by the formation of a blue color at the zone of contact.

37

*Jorissen Method.*¹⁵

Suspend the residue from the ether extract obtained as directed above in about 5 cc. of water; add 2-4 cc. of an approximately 10% solution of mercuric nitrate, and heat for 5-10 minutes on the water bath. In the presence of sucrol a violet blue color is formed, which is changed to a deep violet on the addition of lead peroxid.

FORMIC ACID.

*Quantitative Method.*¹⁶

38

REAGENTS.

(a) *Sodium acetate solution.*—Dissolve 50 grams of dry sodium acetate in sufficient water to make 100 cc. and filter.

(b) *Mercuric chlorid reagent.*—Dissolve 100 grams of mercuric chlorid and 150 grams of sodium chlorid in sufficient water to make 1 liter and filter.

(c) *Tartaric acid.*

(d) *Barium carbonate.*

39

APPARATUS.

The apparatus required (Fig. 6) consists of a steam generator (*S*), a 300 cc. flask (*A*) in which the sample is placed, a 500 cc. flask (*B*), containing a suspension of barium carbonate, a spray trap (*T*), a condenser, and a 1 liter graduated flask (*C*). The tip of the tube (*D*), leading into (*B*), consists of a bulb containing a number of small holes to break the vapor into small bubbles.

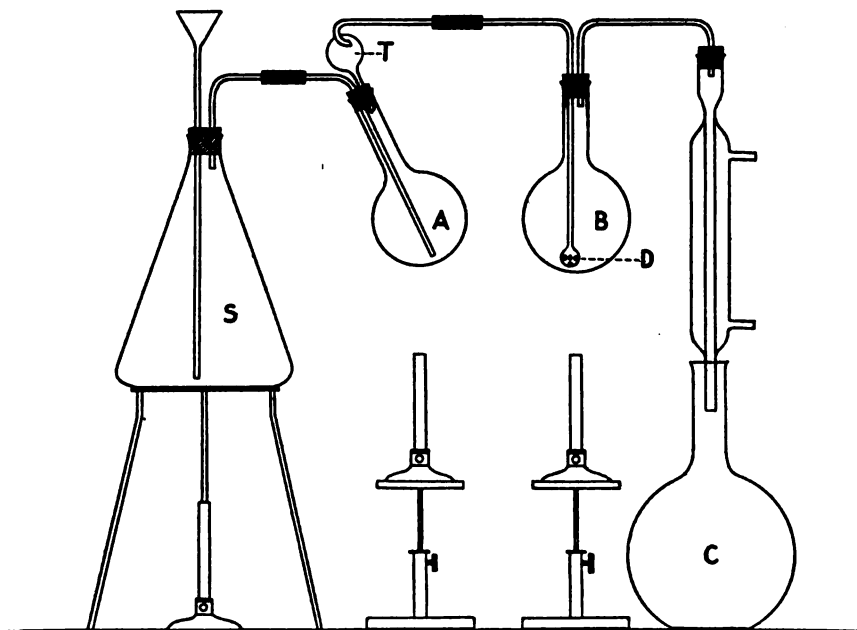


FIG. 6. APPARATUS FOR DETECTION OF FORMIC ACID.

40

DETERMINATION.

For thin liquids like fruit juices, use 50 cc. For heavy liquids and semi-solids like sirups and jams, use 50 grams diluted with 50 cc. of water. Place the sample in the flask (A), add 1 gram of tartaric acid, and connect as shown in Fig. 6, the flask (B) having been charged previously with a suspension of 2 grams of barium carbonate in 100 cc. of water. If much acetic acid is present, sufficient barium carbonate must be used so that at least 1 gram remains at the end of the operation. Heat the contents of flasks (A) and (B) to boiling and distil with steam from the generator (S), the vapor passing first through the sample in flask (A), then through the boiling suspension of barium carbonate in (B), after which it is condensed, and measured in the graduated flask (C). Continue the distillation until 1 liter of distillate is collected, maintaining the volume of the liquids in the flasks (A) and (B) as nearly constant as possible by heating with small Bunsen flames, and avoiding charring of the sample in the flask (A). After 1 liter of distillate has been collected, disconnect the apparatus and filter the contents of flask (B) while hot, washing the barium carbonate with a little hot water. The filtrate and washings should now measure about 150 cc. If not they should be boiled down to that volume. Then add 10 cc. of the sodium acetate, 2 cc. of 10% hydrochloric acid, and 25 cc. of the mercuric chlorid reagent. Mix thoroughly and immerse the container in a boiling water bath or steam bath for 2 hours. Then filter on a tared Gooch, wash the precipitate thoroughly with cold water and finally with a little alcohol. Dry in a boiling water oven for 30 minutes, cool, weigh, and calculate the weight of formic acid present by multiplying the weight of the precipitate by 0.0375. If the weight of mercurous chlorid obtained exceeds 1.5 grams, the determination must be repeated, using more mercuric chlorid reagent or a smaller amount of sample. A blank

test should be conducted with each new lot of reagents employed in the reduction, using 150 cc. of water, 1 cc. of 10% barium chlorid solution, 2 cc. of 10% hydrochloric acid, 10 cc. of the sodium acetate, and 25 cc. of the mercuric chlorid reagent, heating the mixture in a boiling water bath or steam bath for 2 hours. The weight of mercurous chlorid obtained in this blank test must be deducted from that obtained in the regular determination.

BIBLIOGRAPHY.

- ¹ J. Ind. Eng. Chem., 1910, **2**: 24.
- ² Z. Nahr. Genussm., 1910, **19**: 137; C. A., 1910, **4**: 1523.
- ³ U. S. Div. Chem. Bull. 51, p. 113.
- ⁴ Sutton. Volumetric Analysis. 10th ed., 1911, p. 95.
- ⁵ Z. Nahr. Genussm., 1902, **5**: 353.
- ⁶ Analyst, 1895, **20**: 155.
- ⁷ Ann. di farmacoterapia e chim., 1898, **27**: 97; Chem. Zentr., 1898, (1), 1152; 1902, (1), 1076; J. Soc. Chem. Ind., 1898, **17**: 697; Chem. Ztg., 1902, **26**: 246; Abs. J. Chem. Soc., 1902, **82**: 367.
- ⁸ Service de Surveillance des Aliments en Belgique, through Bul. soc. chim. belg., 1897-8, **11-12**: 211; Abs. Analyst, 1897, **22**: 282.
- ⁹ Chem. News, 1905, **91**: 39; Ann. Rept. Mass. State Board of Health, 1905, p. 498.
- ¹⁰ Mon. Sci., 1895, (4), **9**: 324.
- ¹¹ U. S. Div. Chem. Bull. 13, (8), p. 1032.
- ¹² Mon. Sci., 1893, (4), **7**: 842.
- ¹³ Compt. rend., 1893, **117**: 796.
- ¹⁴ Z. anal. Chem., 1896, **35**: 104.
- ¹⁵ Ibid., 628.
- ¹⁶ Biochem. Z., 1913, **51**: 253.



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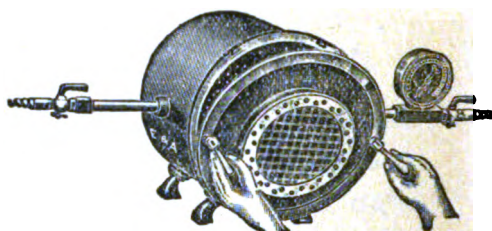
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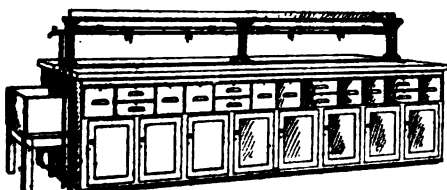
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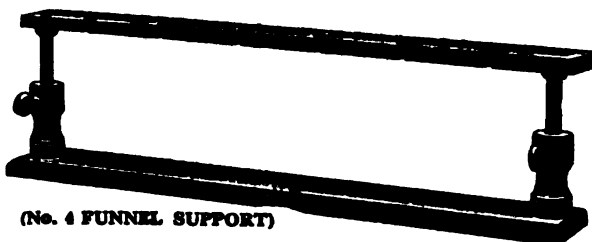
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PROCEEDINGS OF THE THIRTY-SECOND ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, 1915.

FIRST DAY.

MONDAY—MORNING SESSION.

The thirty-second annual convention of the Association of Official Agricultural Chemists was called to order by the President, C. H. Jones, of Burlington, Vt., on the morning of November 15, 1915, at the Raleigh Hotel, Washington, D. C.

REPORT ON WATER.

THE SEPARATION AND DETERMINATION OF CALCIUM AND STRONTIUM.

By W. W. SKINNER (Bureau of Chemistry, Washington, D. C.), *Referee*.

As a result of the experimental work reported by the referee last year, the Stromeier-Rose method for the separation and determination of calcium and strontium was modified by substituting for cold water heretofore used a 1% solution of ammonium oxalate for washing the precipitate of the mixed oxalates. As shown by Holleman, Kohlrausch, Richards and others, the solubility of calcium oxalate in cold water and also in hot water is so low that it may be disregarded as a factor of accuracy in ordinary analytical work. The error in the determination of calcium, due to the solubility of the oxalate, should not be in excess of 1 mg. of calcium when the quantity of precipitate is such that 200 cc. or less of wash water are required.

Strontium oxalate, however, as shown by the experimental work reported last year, is quite soluble in hot water and sufficiently soluble in cold water to cause serious error in the determination of strontium. This is particularly true when relatively small amounts of strontium are to be separated from large amounts of calcium, requiring a greater amount of washing of the precipitate. The method was further modified by providing for 2 extractions of the mixed nitrates with the ether-alcohol mixture, which increased the quantity of solvent used.

The method as modified is as follows:

DETERMINATION OF CALCIUM AND STRONTIUM.

Calcium.

Combine the 2 filtrates obtained from the precipitates of iron and aluminium, concentrate to 150–200 cc. and to this solution, containing not more than 0.6 gram of calcium, calculated as calcium oxid, or 1 gram of magnesium, calculated as magnesium pyrophosphate, add 1–2 grams of oxalic acid and sufficient hydrochloric acid to clear the solution. Heat to boiling and neutralize with ammonium hydroxid, stirring constantly. Add ammonium hydroxid in slight excess and allow to stand for 3 hours in a warm place. Filter off the supernatant liquid and wash the precipitate once or twice by decantation with 1% ammonium oxalate solution. Dissolve the precipitate in hydrochloric acid, dilute to 100–200 cc., add a little oxalic acid, and precipitate as above. After standing for 3 hours, filter, wash with the ammonium oxalate solution as before, dry, ignite, heat over a blast lamp, and weigh as calcium and strontium oxids. Subtract from this weight the weight of strontium oxid equivalent to the strontium sulphate found. The difference is the weight of calcium oxid. Calculate to calcium.

As a check on the calcium oxid, evaporate to dryness the combined filtrates from the strontium nitrate, dissolve the calcium nitrate in water, precipitate as oxalate, filter, wash, ignite and weigh as calcium oxid.

Strontium.

Dissolve in dilute nitric acid the oxids obtained in the determination of calcium and test with the spectroscope for strontium. If strontium is present, transfer the nitric acid solution to a small Erlenmeyer flask. Evaporate nearly to dryness over a low flame and heat in an air bath at 150°–160°C. for 1 or 2 hours after the water is evaporated. Break up the dried material with a stirring rod, add 10–15 cc. of a mixture of equal parts of absolute alcohol and ether to dissolve the calcium nitrate. Cork the flask and allow to stand with frequent shaking for 2 hours or longer. Decant the solution through a 5.5 cm. filter, preserving the filtrate. Wash the residue several times by decantation with small portions of ether-alcohol solution. Dry the residue and the filter paper and wash the filter paper repeatedly with small portions of hot water, collecting the filtrate in the flask containing the main portion of the strontium nitrate residue. Add 1 or 2 drops of dilute nitric acid, evaporate, dry, pulverize, and treat with 10–15 cc. of ether-alcohol mixture as before. Cork the flask and let stand for about 12 hours with occasional shaking. Filter, wash with ether-alcohol mixture until a few drops of the filtrate evaporated on a watch glass leave practically no residue. Dry the paper and precipitate. Dissolve the strontium nitrate in a few cc. of hot water. Add a few drops of sulphuric acid, then a volume of alcohol equal to the volume of the solution and allow to stand for 12 hours. Filter, ignite, weigh as strontium sulphate and calculate to strontium. Test spectroscopically for absence of calcium.

A copy of this method, together with the sample specially prepared for the purpose, was sent to 10 chemists who had signified a desire to undertake the coöperative work and reports have been received from 6 of them.

The solution prepared for the coöperative work contained in the aliquot taken for examination 203 mg. of calcium and 21.8 mg. of strontium.

Results of coöperative work on calcium and strontium.

ANALYST	CALCIUM BY DIFFERENCE	CALCIUM DIRECT	STRONTIUM
	<i>Mg. in 10 cc.</i>	<i>Mg. in 10 cc.</i>	<i>Mg. in 10 cc.</i>
H. P. Corson, Illinois State Water Survey, Urbana, Ill.	200.9	198.6	21.9
O. B. Winter, Agricultural Experi- ment Station, East Lansing, Mich.	204.6 203.9	197.1 196.1	17.1 16.9
E. L. Griffin, Department of Agri- culture, Washington, D. C.	203.2 203.5 203.4	201.5 200.1 203.2	18.1 18.1 17.9
P. L. Hibbard, Agricultural Experi- ment Station, Berkeley, Cal.	203.2 202.3 202.8 202.9	lost lost lost 201.8	20.0 21.9 21.0 21.0
J. W. Sale, Bureau of Chemistry, Washington, D. C.	203.9 203.8 202.8 203.7	202.6 203.2 202.9 202.7	19.1 18.8 19.0 19.6
W. F. Baughman, Bureau of Chem- istry, Washington, D. C.	204.1 203.7 204.3	202.1 202.1 201.9	19.3 20.1 20.3
Maximum.....	204.6	203.2	21.9
Minimum.....	200.9	196.1	16.9
Average.....	203.3	201.1	19.4
Theory.....	203.0	203.0	21.8

By referring to the table it will be noted that the average of the 17 results reported for calcium by difference varies from theory by only 0.3 mg. or 0.15%; while the difference between the highest and the lowest result is only 3.7 mg. These results are as good as could be expected and confirm the observations of the referee upon the results reported in 1913 to the effect that the method for the separation of calcium from strontium and the determination of calcium by the indirect method leaves little to be desired. The calcium direct is slightly low, the average for the 14 results reported being 1.9 mg. or approximately 0.93% below theory. The cause of the low results may be due to an imperfect extraction by the ether-alcohol mixture of the calcium nitrate from the mixed calcium and strontium nitrates but, if so, it apparently does not affect the final result for strontium.

The results reported for strontium while uniformly slightly lower than theory are fairly satisfactory. The results reported by the 6 analysts at this time are fairly concordant and much superior to the results reported in 1913. The work this year and that of previous years shows conclusively that the separation of the calcium and strontium nitrates

by the 2 or more treatments with the ether-alcohol mixture is quite satisfactory, and also that the modified method, substituting a 1% ammonium oxalate solution for cold water in the washing of the mixed oxalates, has eliminated the error in the determination of strontium due to the solubility of strontium oxalate in washing. And while it must be admitted the method gives slightly low results for strontium, due probably to the fact that strontium does not completely precipitate as oxalate,—considerably less so than does calcium,—the results of the investigation and study of the method extending over a period of 4 years are such as to warrant the Association in adopting the method as official and it is so recommended by the referee. The method is presented for its first reading for final adoption as provided in the By-Laws of the Association.

DISCUSSION.

Mr. Skinner: The Committee on Editing Methods of Analysis, Mr. Doolittle, Chairman, early in the year asked the referee to review the methods for the analysis of water. This review showed our methods to be incomplete in that certain standard methods in general use had never been proposed for coöperative work by any referee and had therefore never been adopted as official or provisional methods by the Association, and furthermore, that certain new methods had been substituted for older methods that were not at the same time repealed. It appeared advisable, since the methods were to be printed in the JOURNAL, that such standard methods be included for the sake of completeness, even though they had not been adopted by the Association. These methods were inserted therefore and described as "tentative" to distinguish them from the official and provisional methods. The determinations involved are: turbidity, color, odor, oxygen required (when the chlorin content of the sample is high), dissolved oxygen, specific gravity, hydrogen sulphid, free carbon dioxid, temporary hardness, total hardness, permanent or non-carbonate hardness.

It seemed equally advisable to omit from the JOURNAL those methods which have been superseded, but which have not been officially repealed by the Association. The methods involved are the Pettenkofer method for carbon dioxid and the ammonium sulphate method for barium and strontium.

Under the direction of the referee some time has been devoted to a study of methods for the determination of the radioactivity of waters. The referee has asked Mr. Furber of the Water Laboratory of the U. S. Bureau of Chemistry to present a brief statement of the method and the work.

REPORT ON THE DETERMINATION OF RADIOACTIVITY.

BY F. B. FURBER (Bureau of Chemistry, Washington, D. C.).

The Water Laboratory of the U. S. Bureau of Chemistry has been concerned for some time with the examination of substances claimed to be radioactive, chiefly in connection with the enforcement of the Federal

Food and Drugs Act. The samples have been the so-called "radio-active" bottled mineral waters and various therapeutic preparations.

This paper is based partly on work reported to the U. S. Bureau of Chemistry, January 1, 1915, by Mr. W. D. Collins. Radioactive measurements made by the writer since that time have confirmed Mr. Collins' results and have also established the value of certain other methods. This brief account of the experience of the laboratory is presented merely to bring the matter to the attention of the Association with the hope that eventually uniform procedures for the measurement of radioactivity may be adopted by the Association.

In connection with the routine work, various methods of measurement have been tried and different forms of apparatus used.

The fontactoscope, an apparatus which comprises a gold leaf system and a very large detachable discharging chamber to hold samples of water from which the emanation is separated by shaking, would seem to be unsuited for accurate laboratory measurements, but in the field it is considered serviceable by many¹. The U. S. Bureau of Chemistry has not found field experiments necessary as yet.

The gamma ray electroscope, which is used in measuring quantities of radium as large as a milligram, has not been needed.

An alpha ray electroscope² used by this laboratory for measurement of the radioactivity of substances in solid form makes possible a very rapid and fairly accurate examination of certain drugs and minerals.

An active solid introduced into the discharging chamber gives off chiefly alpha particles which ionize the enclosed air, thus allowing the charge imposed on a gold leaf to escape rapidly. The rate of fall of the leaf is noted just as with an emanation electroscope.

The electroscopes most used in this laboratory are of the emanation type and resemble one made by Charles W. Cook (Manchester, England) after the pattern of Rutherford³. They were made according to specifications which allowed for some modifications of the original. The comparatively small number of samples to be examined made it unnecessary to use an interchangeable electroscope of the kind described by Lind⁴. Also, since in many samples no radioactivity could be detected, it seemed better to have the measuring apparatus in complete fixed units.

For making accurate measurements of low radioactivity the Water Laboratory has followed the generally used method, which is to separate the radium emanation from the substance to be examined and then, by

¹ Engler, Sieveking and Koenig. Chem. Ztg., 1914, 38: 425-7, 446-50; Physik. Z., 1914, 15: 441-7.

² Rutherford. Radioactive Transformations. 1906, p. 28.

³ Rutherford. Radioactive Substances and Their Radiations. 1913, p. 90.

⁴ J. Ind. Eng. Chem., 1915, 7: 406-10.

means of an electroscope, to compare the effect of this with the effect of emanation from a known amount of radium.

If the sample to be tested is a mineral water or solution, but little treatment is needed before separating the emanation by boiling. Soluble solids are dissolved in water or acids. Insoluble substances are fused with mixed sodium and potassium carbonates. This fusion is treated with dilute sodium carbonate solution and the residue remaining after filtration is dissolved in acid. This acid solution and the alkaline extract, which is now acidified, are placed in separate flasks, boiled and sealed. After about 4 days, half the emanation has formed and the gases containing it are collected. Boltwood's apparatus¹ for separating and collecting the emanation has been found very satisfactory. If the solution is boiled for about 20 minutes, the emanation appears to be separated completely.

The Schlundt and Moore apparatus² for collecting emanation by boiling it out, does not work properly unless given very close attention. It is possible also to extract emanation from solutions by bubbling air through while heating³ and this method is often more simple and convenient than others. The gases obtained by boiling the solution are kept for 10 minutes, to allow for the decay of any thorium emanation present, and then transferred through a calcium chlorid drying tube to the discharging chamber of an emanation electroscope. The rate of fall of the charged gold leaf is observed about 3 hours after this. The electroscope is calibrated by measuring the rate of discharge caused by a known amount of emanation formed in a standard radium solution. The solution of radium barium bromid in hydrochloric acid (1.10 sp. gr.) used in the U. S. Bureau of Chemistry was furnished by the U. S. Bureau of Standards.

The electroscopes have been calibrated also by the use of pitchblende, the uranium content of which was determined as described by Brearley⁴. A sample of the ore is dissolved in nitric acid and the emanation collected and passed into the electroscope by Boltwood's method. Correction is made for the emanation lost spontaneously by sealing up a quantity of the ore in a glass tube and at the end of a month transferring the collected emanation to an electroscope by means of a stream of air. The temperature at which the sample is kept has an appreciable effect on the amount of emanation given off by the ore. If time permits, this correction may be eliminated by using the method of solution described by Lind and Whittemore⁵ in which the ore is sealed up for a month in the flask where it is to be dissolved. Then, when the emanation in equilibrium

¹ Phil. Mag., 1905, 6th ser., 9: 599-613; Am. J. Sci., 1904, 4th ser., 18: 378-87.

² J. Phys. Chem. 1905, 9: 320-32.

³ Curie. *Traité de Radioactivité*. 1910, 1: 284.

⁴ Brearley. *The Analytical Chemistry of Uranium*. 1903; see also U. S. Bur. Mines Bull. 70, and J. Am. Chem. Soc., 1914, 36: 2075-8.

⁵ J. Am. Chem. Soc. 1914, 36: 2071-2.

with the ore is all confined, solution in acid gives the total emanation corresponding to the radium present.

In many cases, to save time and manipulation, departure has been made from approved methods given in the literature but a detailed discussion is impossible here. For example, in most of the Water Laboratory's work the extra flask of Boltwood's collecting apparatus has been found unnecessary and a plain 100 cc. Erlenmeyer flask has been used for dissolving certain minerals and boiling strongly active solutions.

There has not been, as yet, occasion for making an extended investigation of thorium preparations. Thorium emanation has so short a life that it must be measured by passing it in a continuous stream through an electroscope. The method of Mache and Bamberger¹ seems to be the most satisfactory of any in the literature.

In dealing with mineral waters and most medicinal preparations this laboratory reports radioactivity in millimicrocuries (billionths (10^{-9}) of a curie)² per liter of solution or gram of substance. Many results in the literature have been expressed in mache or electrostatic units, but since so many investigators have not allowed for corrections (chiefly the Duane³ correction) it is hard to place absolute reliance on their results or to make comparisons between results stated in the two different systems. For most purposes, however, the mache unit equals 0.37×10^{-9} curies or the electrostatic unit equals 0.37×10^{-6} curies is accepted as a satisfactory conversion ratio. The Water Laboratory accepts also the uranium-radium ratio of Heinmann and Marckwald⁴. (One gram of uranium equals 332.8×10^{-9} grams of radium.)

In regard to the magnitude of amounts of emanation determined, it may be said that the emanation electroscopes of the Water Laboratory have a limit of sensibility corresponding to 0.005×10^{-9} grams of radium, and will measure easily amounts of emanation corresponding to 20×10^{-9} grams of radium.

Some samples of artificial waters and medicinal preparations have been found to be as radioactive as claimed on the label. Most natural waters, when bottled, lose all their radium emanation after 30 days, because they do not contain dissolved radium salts or insoluble suspended radioactive matter to make them permanently active. Such waters, therefore, and also a number of artificial products, have been found so slightly radioactive at the time of sale that, in comparison with actual doses of radium salts used in medicine, and with the waters of natural springs known for their radioactivity at source, the radioactivity is wholly negligible.

¹ Sitzb. kais. akad. Wiss. Wien. Abt., 1914, 123 (IIa): 334-45.

² One curie is the radioactivity corresponding to one gram of radium element.

³ Compt. rend., 1905, 140: 581-3; 1910, 150: 1421-3.

⁴ Physik. Z., 1913, 14: 303-5.

WATER IN FOODS AND FEEDING STUFFS.

By W. J. McGEE (Food and Drug Inspection Laboratory,
U. S. Custom House, Savannah, Ga.), *Referee*.

The principal object of this year's work was the study of the method of drying without heat in a vacuum over sulphuric acid. The following outline of the method was sent out for trial:

DRYING IN VACUO WITHOUT HEAT.—OPTIONAL.

(This method was first suggested by Mr. Trowbridge at the meeting of the Association in 1909; approved in 1913 for final action as an optional official method in 1914; final action not yet taken.)

Mix the sample thoroughly and weigh about 2 grams by difference from a stoppered weighing bottle into tared crucibles provided with covers which are tared with crucibles. Place 200 cc. of fresh C. P. sulphuric acid in a good 6 inch vacuum desiccator. Put triplicate samples in the desiccator, smear the edges of the latter and the stop-cock with lubricant (a mixture of 3 parts of hard paraffin and 5 of vaseline) and exhaust by means of a vacuum pump. If a pump is not available, place 10 cc. of ether contained in a small beaker in the desiccator and exhaust with a water filter pump. It will be found convenient to interpose between the pump and the desiccator an empty bottle next to the desiccator and a bottle of water following this. Draw the air from the desiccator through the water and turn the desiccator stop-cock at just the instant when the water begins to rise in the tube leading from the empty bottle. Gently rotate the desiccator 4 or 5 times during the first 12 hours to mix the sulphuric acid with the water which has collected as an upper layer. At the end of 24 hours open the desiccator, forcing the incoming air to bubble through C. P. sulphuric acid. If a good vacuum has been maintained the samples are ready for the first weighing. After weighing place in a desiccator with fresh C. P. sulphuric acid and exhaust as before. Rotate the desiccator once or twice during the interval and weigh again at the end of 24 hours, repeating this process of drying in vacuo over sulphuric acid until the weight is constant.

REPORTS OF COLLABORATORS.

W. D. Richardson: Sausage meat (vacuum method).—Constant weight in about 208 hours with a loss in weight of about 1% less than by the heating methods.

D. B. Bisbee: Cheese (vacuum method).—Weight nearly constant eighth day. Loss of weight about 0.4% less than by heating methods.

J. O. Clarke: Cottonseed meal (vacuum method).—Weight practically constant in 9-15 days with loss about the same as in heating methods.

Corn meal.—In vacuum desiccator dried to approximately constant weight in 4 days and the loss was about 0.4% less than by heating methods.

Tomato ketchup.—With water content of 83.11% as indicated by heating at 100°C. in water oven, lost 80.15% in vacuo over sulphuric acid in 5 hours, and 81.21% in 5 days. In another experiment lost 80.53% in 18 hours and 80.72% in 42 hours. Both of these experiments were terminated by accident before constant weight was attained.

Hamburger steak.—In vacuo over sulphuric acid lost 70.42% of its weight in 4 days; by heating methods, 71.09%.

Lean meat.—Ground fine lost 65.56% in 44 hours and 67.10% by heating methods.

Apple jelly.—Lost 21.66% in 48 hours and 27.45% in vacuum oven at 70°C.

Cattle Food Laboratory, U. S. Bureau of Chemistry: Comparison of this method with drying at 100°C. at the house vacuum (average 86.4) gave the following results:

Mixed alfalfa, molasses, corn feed at 100°C.—Average pressure 86.4 mm. Moisture, 3.60%.

At 27°C.—Average pressure 7.5 mm. Moisture 1.89%.

Bran at 100°C.—Average pressure 86.4 mm. Moisture 10.79%.

At 27°C.—Average pressure 7.5 mm. Moisture 10.43%.

Linseed meal at 100°C.—Average pressure 86.4 mm. Moisture 10.86%.

At 27°C.—Average pressure 7.5 mm. Moisture 10.71%.

TABLE 1.
Moisture determinations under varying conditions.
(J. H. Roop, Indiana.)

SUBSTANCE	DRIED 5 HOURS IN HYDROGEN AT 100°C.	DRIED 5 HOURS IN VACUO AT 75°C.	DRIED IN 6 INCH VACUUM DESICCATOR WITH SULPHURIC ACID AVERAGE
	<i>Per cent loss</i>	<i>Per cent loss</i>	<i>Per cent loss</i>
Cottonseed meal.....	8.75	8.10	7.55
Wheat bran.....	11.65	11.27	11.04
Corn germ meal.....	4.82	4.45	4.36
Linseed meal.....	9.97	9.57	9.15
Corn and oats.....	13.01	12.42	12.68
Distillers' grains.....	8.90	7.95	7.64

C. O. Swanson: This method has been in use at the Kansas Agricultural College for several years and is considered very satisfactory. For flour the drying is continued for from 3–5 days. Very moist samples are allowed to remain in the desiccator for 3 weeks. A shaking machine is employed to keep the acid stirred up.

COMMENTS BY THE REFEREE.

It has been found at the U. S. Food and Drug Inspection Laboratory at Savannah that the desiccator acid containing ether and water can be recovered by digesting for about 4 hours over a free flame in a Kjeldahl flask with a drop of mercury. A study of this method from the work of this and previous years seems to show that it can be relied upon when heating the sample is inadmissible; and that it is valuable also when subsequent fat extraction is contemplated and heating would tend to harden the sample too much. For work of this kind in which time is not a factor it may be very convenient to have an official method.

TABLE 2.

Comparison of dehydrating powers of reagents used in desiccators at room temperature of 20°-33°C. and at atmospheric pressure.

SUBSTANCE	TIME	SULPHURIC ACID	CALCIUM CARBID	SODIUM METAL	CALCIUM OXID	CALCIUM CHLORID	SODIUM HYDROXID
	Days	Per cent loss	Per cent loss	Per cent loss	Per cent loss	Per cent loss	Per cent loss
Corn meal ¹ :	1	10.18	10.06	9.54	9.22	7.76
	2	11.13	10.85	10.69	9.84	8.72
	3	11.49	11.22	11.09	10.05	9.21
	4	11.60	11.29	11.03	10.02	9.06
	5	11.74	11.43	11.20	10.10	9.19
	6	11.82	11.54	11.22	10.14	9.28
	7	11.87	11.53	11.30	10.14	9.23
Lean meat, finely ground ² :	1	43.70	36.20	45.23	28.77
	2	65.15	63.22	62.69	56.41
	3	66.04	65.50	63.53	64.08
	4	66.40	65.97	63.89	64.75
	5	66.56	66.10	63.98	64.90
	6	66.65	66.27	64.13	65.12
	7	66.78	66.38	64.23	65.25
	10	66.98	66.62	64.40	65.50
Hamburger steak ³ :	Hours						
	3	9.00	11.93	8.73
	Days						
	1	59.26	62.17	54.74
	2	69.50	69.40	69.15
	3	70.16	69.97	69.82
	4	70.47	70.24	70.14
Tomato paste ⁴ :	1	77.51	40.16	77.10	77.32
	2	77.67	76.40	77.35	77.54
	3	77.98	77.10	77.50	77.64
Glucose and apple jelly ⁵ :	1	18.91	19.59	17.84	16.43
	2	19.62	18.07	16.57
Pure apple jelly ⁶ :	1	19.29	20.44	18.90
	2	20.17	20.84	19.78
	3	21.11	20.19

¹ Vacuum oven 12.60%; in hydrogen 13.51%.

² Loss at 100°C., 67.10%.

³ Loss at 100°C., 71.09%.

⁴ Vacuum oven at 70°C., 78.4%.

⁵ Vacuum oven at 70°C., 23.83%.

⁶ Vacuum oven at 70°C., 27.45%.

It appears from this work and from the experience of previous years, particularly as reported at the meeting in 1913, that calcium carbide is a desiccator reagent nearly as efficient as sulphuric acid and one that is portable. This reagent is good until a large percentage of the lumps have loosened up into powder.

It will be best, however, to continue using sulphuric acid, lime or calcium chloride for precipitates of copper suboxide.

Leaving phosphorus pentoxide out of the question on account of its high cost and its rapid decrease in efficiency, it appears that the 3 best desiccator reagents are sulphuric acid, calcium carbide and calcium oxide, in the order named.

RECOMMENDATIONS.

It is recommended—

(1) That further work be done in comparing the drying of various food and feeding products over sulphuric acid, calcium carbide and calcium oxide, with any other reagents thought desirable, at atmospheric pressure and in a partial vacuum.

(2) That the method of drying without heat over sulphuric acid as outlined above be finally made an official optional method.

DISCUSSION.

P. F. Trowbridge: Mr. Grindley of the University of Illinois has used the method with good results—much better than we have been able to obtain at our Missouri Laboratory. Some materials present difficulties because they dry to a horn-like substance. The referee has not sufficiently emphasized the need for a good vacuum. Seven millimeters of mercury are not enough. A pump is necessary. With a pressure not exceeding 1 mm. it is possible to obtain from 2-10% more moisture from grain than by drying at 102°C.

REPORT ON FOODS AND FEEDING STUFFS.

By G. L. BIDWELL (Bureau of Chemistry, Washington, D. C.), *Referee*.

The coöperative work this year was all in charge of associate referees.

It is recommended—

(1) That the method for the detection of oats bleached with sulphur dioxide¹, be made official.

(2) That the method for determining the acidity of corn², be made official.

(3) That the method for determining the acidity of corn be studied to see if it is applicable to other materials.

(4) That the effect of preliminary drying on the ether extract content of feeds be studied.

(5) That the use of toluol or other suitable substance as a preservative for feed samples be studied.

(6) That the method of Bryan, Given and Straughn³ for the estimation of sugar in feeds be studied and made official.

FEED ADULTERATION.

By C. CUTLER (Agricultural Experiment Station, La Fayette, Ind.),
Associate Referee.

The work during the past year was confined to the following recommendation, approved at the 1914 meeting:

“(1) That samples be sent out for the quantitative determination of

¹ U. S. Bur. Plant Industry Circ. 40.

² Ibid., Bull. 199; U. S. Dept. Agr. Bull. 102.

³ U. S. Bur. Chem. Circ. 71.

adulterants on amounts varying from 5-25 grams to determine the smallest amount necessary to work with to get concordant results."

The following instructions were sent with the samples to 9 prospective collaborators:

INSTRUCTIONS FOR COLLABORATIVE WORK.

Quarter the samples, discard alternate quarters until the residue weighs approximately 25 grams. Procure a 10 gram sample in the same manner. A hand lens may be used in picking out small pieces of adulterants. The use of 20, 30 and 40 mesh sieves aids the work.

Sample No. 1 (wheat bran and screenings).—Determine the total amount of foreign material in 10 and 25 gram portions and identify the same.

Sample No. 2 (linseed meal containing cottonseed meal and hulls).—Determine the total amount of cottonseed meal and hulls in 10 and 25 gram portions.

Sample No. 3 (scratch feed).—Determine the total amount of grit in 10 and 25 gram portions; also the total amount of weed seeds present, identifying the principal ones.

Sample No. 4 (scratch feed containing coarse grit).

Sample No. 5 (wheat bran containing screenings).

RESULTS OF COLLABORATIVE WORK.

Per cent of adulterants found.

ANALYST	FOREIGN MATERIAL IN SAMPLE NO. 1		COTTONSEED MEAL AND HULLS IN SAMPLE NO. 2		WEED SEEDS IN SAMPLE NO. 3		GRIT IN SAMPLE NO. 3		GRIT IN SAMPLE NO. 4			SCREENINGS IN SAMPLE NO. 5	
	10 gram sample	25 gram sample	10 gram sample	25 gram sample	10 gram sample	25 gram sample	10 gram sample	25 gram sample	10 gram sample	25 gram sample	50 gram sample	10 gram sample	25 gram sample
A. W. Clark and O. C. McCreary, Agricultural Experiment Station, Geneva, N. Y. }	4.2	3.2	5.0	4.1	32.1 ¹	32.9 ¹	22.6	24.0
B. H. Silberberg, Bureau of Chemistry, Washington, D.C. }	7.5	7.6	19.1 24.4	21.5
G. W. Hoover and F. G. Smith, Transportation Building, Chicago, Ill. }	5.1	4.2	0.58 ²	0.45 ²	33.5 ¹	33.6 ¹	23.6	21.1
P. H. Smith and Geo. H. Chapman, Agricultural Experiment Station, Amherst, Mass. }	2.1	2.5	15.4 ³	14.0 ³	21.9	23.1
Carleton Cutler, Agricultural Experiment Station, La Fayette, Ind. }	5.8	5.7	6.8	6.6	25.9	24.1	24.3 22.8	23.0 22.4	5.0	7.4	6.0 6.4	2.7	2.7

¹ Includes millet.

² Cottonseed hulls. Estimation of cottonseed meal present based on microscopical examination, 2-3%.

³ Includes timothy, but not shrivelled alfalfa and clover seeds.

The greatest variation in these results is in the determination of grit in Sample No. 3. However, the average of the 10 gram samples compares favorably with the average of the 25 gram samples.

Sample No. 4, scratch feed, contained very coarse limestone grit. Determinations were made on carefully quartered 25 and 50 gram samples. The 2 determinations on the 50 gram sample gave 6.0 and 6.4% grit, showing a variation of only 0.4%; while the 10 gram sample gave 5%, and the 25 gram sample 7.4%. Thus, the larger sample gives more concordant results on feeds containing coarse grit.

The variation in percentage of weed seeds found by different analysts in scratch feed No. 3, is due to the fact that all small seeds, including millet, were determined by 2 of the collaborators reporting. The determinations on the 10 and 25 gram samples agree very closely, the average variation on 3 determinations being only 0.3%.

The determinations of screenings in Sample No. 1, and cottonseed meal and hulls in Sample No. 2, are fairly concordant on 10 gram and 25 gram samples.

The results on Sample No. 5, wheat bran and screenings, made on 10 and 25 gram samples show identical percentages of foreign materials.

RECOMMENDATIONS.

Based upon the facts brought out in this year's results, together with experience obtained during the past several years, it is recommended—

(1) That the following method for the approximate estimation of foreign material, excluding grit, in feeding stuffs, be adopted as a provisional method:

PREPARATION OF SAMPLE.

Thoroughly mix the original unground sample, quarter, discard diagonally opposite quarters until the remaining 2 quarters weigh 10 grams.

DETERMINATION.

Separate the 10 gram sample by means of 20, 30, 40 and 50 mesh sieves. With the aid of a hand lens, or other magnifying instrument, pick out foreign materials; first from the finest portion, and then from the next finest, and so on in order until all portions have been examined. Combine the foreign materials separated and weigh.

(2) That the size of sample of scratch and poultry feeds necessary to get concordant results on quantitative grit determinations be further investigated.

(3) That the following recommendations of 1914 be studied during the coming year:

"3. That methods for the detection of peat dried at high temperatures in feed stuffs be investigated," and "4. That the maximum percentage of foreign materials permissible in mill by-products be investigated".

REPORT ON CRUDE FIBER.

By C. K. FRANCIS (Agricultural Experiment Station, Stillwater, Okla.),
Associate Referee.

The work on crude fiber has been confined to a study of the official method, with linen and asbestos filters, and the one filtration method, using paper as a filter.

Samples of fat-free kafir corn and cottonseed meal were sent to the cooperating chemists with the following instructions:

INSTRUCTIONS TO COLLABORATORS.

Pass the sample through a 20 mesh sieve. Make all determinations in triplicate and report the time required for the analysis under each method.

Weigh portions of 2 grams on hard filter papers and extract 4 times with ether, or use the residue from the crude fat determination.

DETERMINATION OF CRUDE FIBER.

Method 1.—Official¹.

Make the first filtration through linen and the second through asbestos. Report fineness of the linen in number of threads to the inch, counting both ways. Dry and incinerate as directed under Method 2. Make blank determinations to show any loss of weight of the asbestos.

Method 2.—One Filtration Through Paper.

Brush carefully the dry, fat-free residue into an 800 cc. lipless beaker. Add 200 cc. of boiling 1.25% sulphuric acid and boil for 30 minutes, using round-bottomed flasks filled with cold water as condensers placed on top of the beakers. The flasks should fit closely if the beakers are round and have well-formed rims. (Kjeldahl flasks are used in the associate referee's laboratory.) Direct a gentle blast of air into the beaker when the foaming becomes serious. At the end of 30 minutes add 200 cc. of boiling 3.52% sodium hydroxid and continue the boiling for another 30 minutes.

A Büchner funnel, outside diameter 10.5 cm., is prepared with a 9 cm. S. & S. No. 575 hard filter paper. The paper should be moistened with water and pressed well into place, so that there will be no holes uncovered and no open channels left about the edges. It is advisable to place a glass filter plate over the paper, to protect it and to prevent excessive packing of the fiber.

After the 30 minutes alkali boiling, filter rapidly with the aid of suction, wash with boiling water, then with a 1.25% solution of concentrated hydrochloric acid (14 cc. made up to 500 cc. with water), until the washings are acid. Much time may be saved if the liquid is set aside for about 2 minutes to allow the fine material to settle so that the clear liquid may be decanted. Wash with hot water until free from chlorids, and finally wash the filter several times with 80% alcohol. Remove the paper from the funnel. Transfer the residue to a Gooch crucible with the aid of a spatula and a small amount of 80% alcohol. Rub the paper lightly with the finger to detach the last traces of the fiber residue.

Place the crucible in an oven heated to 105°–110°C. for from 2–6 hours, cool and weigh. The material may become dry in 2 hours but when large quantities of fiber

¹ U. S. Bur. Chem. Bull. 107, Rev., p. 56.

are present a longer heating will be necessary. Heat for 30 minutes before weighing the second time.

Partially cover the crucible and incinerate at a low red heat for 15 minutes or until a white or light gray ash is obtained. Cool and weigh. The loss of weight is crude fiber.

RESULTS OF COLLABORATIVE WORK.

TABLE 1.
Determination of crude fiber and time required.

ANALYST	KAFIR CORN				COTTONSEED MEAL				NUMBER OF THREADS TO THE INCH OF FILTER- ING CLOTH
	Official Method		Proposed Method		Official Method		Proposed Method		
	per cent	hours	per cent	hours	per cent	hours	per cent	hours	
O. C. Smith, Agri- cultural Experi- ment Station, Stillwater, Okla.	1.73	3	1.77	2	10.38	3	11.23	2	60 × 57
	1.70	1.69	10.20	10.59	
	1.70	1.64	10.44	10.67	
	Average.....	1.71	1.70	10.34	10.63	
D. G. Morgan, Ag- ricultural Ex- periment Sta- tion, Stillwater, Okla.	1.81	3½	1.62	3	10.32	3½	11.65	3	60 × 57
	1.80	1.78	10.42	10.80	
	1.83	1.64	10.24	10.61	
	Average.....	1.81	1.68	10.33	10.70	
G. W. Roark, Ag- ricultural Experi- ment Station, College Station, Tex.	1.80	1.63	10.11	10.81	6	42 × 42
	1.77	1.10	10.37	
	1.73	1.56	10.25	10.44	
	Average.....	1.76	1.43 ¹	10.24	10.62	
A. S. Wells, Dairy and Food Com- mission, Port- land, Ore.	1.75	8	1.90	7	9.57	10	11.01	16	92 × 92
	1.80	1.89	9.60	
	1.75	1.94	9.80	10.82	
	Average.....	1.76	1.91	9.65 ¹	10.91	
P. F. Trowbridge, Agricultural Ex- periment Sta- tion, Columbia, Mo.	1.52	10	1.64	7	10.05	10	11.16	7	68 × 71
	1.53	1.85	10.14	11.01	
	1.75	1.81	10.18	11.25	
	Average.....	1.60	1.76	10.12	11.14	
G. P. Walton, Bu- reau of Chemis- try, Washington, D. C.	1.70	4½	1.54	9	10.87	4½	10.17	9	42 × 46
	1.70	1.50	10.41	9.41	
	1.79	1.53	10.33	10.07	
	Average.....	1.73	1.52	10.53	9.88 ¹	

¹ Omitted from corrected general average.

TABLE 1.—Continued.

ANALYST	KAFIR CORN				COTTONSEED MEAL				NUMBER OF THREADS TO THE INCH OF FILTER- ING CLOTH
	Official Method		Proposed Method		Official Method		Proposed Method		
	per cent	hours	per cent	hours	per cent	hours	per cent	hours	
Jas. W. Kellogg, Department of Agriculture, Har- risburg, Pa.	1.62	3.06	9.59	14.07	75 × 75
	1.57			9.59			
	1.60			9.63	12.65	
	Average.....	1.59	3.06 ¹	9.60 ¹	13.36 ¹	
W. D. Richardson, Swift and Com- pany, Chicago, Ill.	1.47	3	1.55	2	9.13	4	10.03	4-5	87 × 87
	1.60	1.58	9.48	10.37	
	1.56	1.40	9.78	10.55	
	Average.....	1.54	1.51	9.46 ¹	10.31	
F. G. Smith, Trans- portation Build- ing, Chicago, Ill.	1.64	1½	1.91	1½	10.64	1½	11.19	4-8	58 × 54
	1.70	1.93	10.75	11.30	
	1.74	1.96	11.01	11.99	
	Average.....	1.69	1.93	10.80	11.24 ¹	
D. T. Evans, Fort Worth, Tex.	1.69	1	1½	10.10	1½	2	80 × 100
	1.67	9.95	
		9.42	
	Average.....	1.68	9.82 ¹	
J. H. Roop, Agri- cultural Experi- ment Station, La Fayette, Ind.	1.65	3	1.82	5	9.87	3	10.32	5	75 × 76
	1.63	1.82	9.81	9.92	
	1.60	1.75	9.96	10.30	
	Average.....	1.63	1.80	9.88 ¹	10.18 ¹	
General average ... Corrected general average.	1.68	1.83	10.07	10.90	
	1.68	1.72	10.39	10.72	

¹ Omitted from corrected general average.

COMMENTS OF ANALYSTS.

G. W. Roark: Method 2 was shorter and easier than the regular (Official) method but there was difficulty in filtering through paper; also, it is believed, there is some loss around the edges of the paper.

A. S. Wells: Fine material passes through the linen during the first filtering. This is the main objection to the official method. The proposed method worked fairly well with the sample of kafir corn, but the sample of cottonseed meal packed so hard that it could not be removed without taking off some of the filter paper.

P. F. Trowbridge: After trying a great number of samples of muslin and linen we secured a very fine grade of muslin, 98 x 102 threads per inch. The best grade

of linen we were able to secure ran 68 x 71 threads to the inch. Furthermore, the particles of fiber adhere much more to the linen threads than to the muslin threads.

Considerable difficulty was experienced in filtering through asbestos. The proposed method was more rapid than the official method, but the filtering was slow and there was difficulty in transferring the fiber from the paper to the Gooch crucible. Whenever any modification of the Sweeney method has been tried with single filtration higher results have always been obtained.

G. P. Walton: As pointed out in last year's report by this laboratory on crude fiber, this one filtration method so modifies the regular method as to make it extremely improbable that results from the 2 methods can be reconciled. Besides the question of the redissolving of substances precipitated, on making the acid liquid alkaline, there is the real objection to digesting with twice the volume of 1.25% sodium hydroxid solution specified in the official method.

The transfer of the fiber residuum is a tedious operation, and difficulty was experienced in obtaining all of it without including fiber from the filter paper.

Directions to wash finally with strong alcohol should be incorporated in the official method.

J. W. Kellogg: Proposed method entirely unsatisfactory when compared with the official method.

W. D. Richardson: With such samples as cottonseed meal it was found quite difficult to pass 400 cc. through the same filter, as the pores soon became clogged and made the filtration and washing unsatisfactory. Kafir corn gives good results by this method and is easy to handle.

F. G. Smith: Method *§* required from 4-8 hours to make the digestion and filtration, in one case it being practically impossible to complete the filtering.

D. T. Evans: The filtration through paper was so difficult that asbestos was substituted.

J. H. Roop: Great difficulty was encountered in filtering the alkaline solution through the paper and in transferring the fiber from the filter paper without including some fiber of the filter paper.

EXPERIMENTS BY THE ASSOCIATE REFEREE.

It may be of interest to mention some additional work on crude fiber. The data presented in Table 2 show the results obtained with 3 kinds of filters when but one filtration was made.

TABLE 2.

Determination of crude fiber in cottonseed meal by the one filtration method using different filters.

FILTERS	SEPARATE DETERMINATIONS			AVERAGE
	per cent	per cent	per cent	per cent
Linen.....	8.57	8.58	8.54	8.56
Hard paper.....	9.54	9.53	9.37	9.48
Asbestos.....	10.87	10.81	10.84

Portions of cottonseed meal from the same sample were examined in a similar manner, observing the directions of the official method.

TABLE 3.

Determination of crude fiber in cottonseed meal by the official method using different filters.

FILTER USED		SEPARATE DETERMINATION			AVERAGE	REMARKS
After acid digestion	After alkali digestion	per cent	per cent	per cent		
Linen	Linen	8.44	8.16	7.96	8.19	No suction; residue washed into a Gooch crucible.
Linen	Asbestos	8.56	8.82	8.69	Asbestos filter into a Gooch crucible, i.e., small amount asbestos used.
Linen	Asbestos	8.96	8.79	9.02	8.92	Asbestos filter in a Hirsch funnel, i.e., large amount asbestos.
Paper	Paper	9.63	9.53	9.51	9.56	S. & S. No. 575 paper in a Büchner funnel.

It will be observed from the data in Tables 2 and 3 that the determinations with a given filter, when considered alone, appear satisfactory, but when comparisons of the groups are made, the results do not agree.

Determinations in addition to those here reported have shown that the loss with linen depends upon the number of times the mixture is filtered through it, thus demonstrating that there is a loss of crude fiber.

The crude fiber determinations in which filter papers were used appear to be the most uniform for both methods. The paper filters certainly do not permit any solid matter to pass into the filtrate, and they offer a uniform filter which may be easily obtained and from which the residue can be readily transferred.

Some determinations were made in which asbestos was used for both filtrations according to the official method, but the large quantity of material added to the second filter, in this way, caused the filtration to proceed very slowly.

Portions of filter paper, S. & S. No. 597, were ground so as to pass a 40 mesh sieve. This material was intended to be used for testing the efficiency of different filters, but the residue obtained, after passing through the entire official method for crude fiber, showed so great a loss, that it became necessary to ascertain where the loss occurred. The final procedure involved digestion of separate samples in 1.25% sulphuric acid and in 1.25% sodium hydroxid, and in both reagents as outlined in the method for one filtration.

The results of this test are presented in Table 4.

TABLE 4.

Action of crude fiber reagents on pure filter paper.

PROCEDURE	FIBER RECOVERED BY USE OF PAPER FILTERS		FIBER RECOVERED BY USE OF ASBESTOS FILTERS	
	Original basis	Ash plus water-free basis	Original basis	Ash plus water-free basis
	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>
Acid digestion only:	93.90	99.42	101.15	107.14
	95.03	100.62	102.53	108.56
	94.56	100.11	97.38	103.10
	Average.....	94.49 100.05	100.35	106.27
Alkali digestion only:	91.33	96.70	98.06	103.86
	92.27	97.72	98.52	104.38
	91.78	97.17	98.74	104.59
	Average.....	91.79 97.20	98.44	104.28
Acid and alkali, 1 filtration:	64.91	68.75	71.50	75.71
	64.67	68.47	74.71	79.13
			73.00	77.30
	Average.....	64.79 68.61	73.07	77.38
Acid and alkali, 2 filtrations:	65.02	68.85		
	65.58	69.18		
	64.26	68.05		
	Average.....	64.95 68.69

The action of the acid is apparently negligible, and the alkali does not seem to dissolve a very large quantity. However, the material digested in both acid and alkali showed a very large loss, approximately 31% by both methods. The asbestos filter gave higher results in all tests.

CONCLUSIONS.

The results reported this year indicate that hard filter paper, used in the manner suggested, is not satisfactory. All analysts obtained good results for kafir corn by the official method and the returns on the same sample tested by the one filtration method were encouraging. However, the showing for the sample of cottonseed meal was not so good and indicates a need for further study of crude fiber methods. A number of the chemists did not report the loss of weight due to the asbestos, but those reporting indicated that an error of from 0.05-0.20% plus, may be introduced. Results showing much higher losses were reported by several chemists.

THE DETERMINATION OF CRUDE FIBER¹.

BY C. E. MANGELS AND P. F. TROWBRIDGE (Agricultural Experiment Station, Columbia, Mo.).

In laboratories where a great many crude fiber determinations are made, it is important that the method used be as free as possible from tedious manipulations. As in all empirical determinations, however, it is important that the methods followed should be the same as in other laboratories until it has been shown that slight modifications do not affect the accuracy of the results.

In the collaborative work, the referee's samples were tested by methods which have been used in the Missouri Agricultural Experiment Station. The method in use this year and which seems to give the most satisfactory results is as follows:

Transfer the residue from the ether extraction to a lipless beaker, add 200 cc. of cold 1.25% sulphuric acid and make a mark on the beaker to indicate the height of the liquid. Heat rapidly to boiling the contents of the beaker and continue the boiling for exactly 30 minutes. Filter rapidly the contents of the beaker through a muslin filter and wash the fiber free from acid with hot water. Place the original beaker under a funnel and transfer the fiber from the muslin to the beaker, using a spatula and a stream of 1.25% sodium hydroxid from a wash bottle. Then fill the beaker to the mark with 1.25% sodium hydroxid, heat rapidly to boiling the contents of the beaker and boil for exactly 30 minutes. Filter the hot alkaline solution through the same muslin filter, wash the beaker twice with hot water and transfer the washings to the filter. Then wash the fiber on the filter once with 1.25% hydrochloric acid and then thoroughly with hot water to remove all acid and chlorids. Transfer the bulk of the fiber from the muslin to a prepared Gooch crucible, spreading the fiber on the sides of the crucible so as to avoid clogging the filtration. Place the original beaker under the funnel and wash the last traces of the fiber from the muslin into the beaker by means of a stream of hot water. Connect the Gooch crucible with suction and transfer the fiber in the beaker to that in the crucible, by the aid of hot water and a policeman. Wash finally the fiber in the Gooch crucible with alcohol and dry to constant weight at 102°C., then ignite and weigh again to determine the fiber by difference.

The writers have tested a great many samples of linen and muslin to secure a satisfactory medium for filtering and have finally selected and secured a quantity, sufficient for several years, of close-woven muslin having 98 x 102 threads to the inch. The best linen which could be secured had 68 x 71 threads to the inch.

The method just described differs from the regular official method in that both the acid and alkali solutions are added to the sample cold, thereby making the contact with the sample 30 minutes in addition to the time required to heat the 200 cc. of solution to the boiling point. This, if anything, should give lower results than by the regular official method.

¹ Presented by P. F. Trowbridge.

The results are shown under Method C in the following table and are slightly higher than results obtained by the regular official method (A). The writers have at different times tried the Sweeney method (single filtration) with various modifications, hoping to get a rapid method which was at the same time comparable with the regular official method.

In this method, the results of which are reported under (D) in the following table, 200 cc. of boiling 1.25% sulphuric acid were added to the fiber and the boiling continued for exactly 30 minutes. Two hundred cc. of boiling 3.52% sodium hydroxid were added immediately to this solution and the boiling continued for another 30 minutes. Rapid filtration was then made through the muslin filter. The beaker was washed twice with hot water, the washings being transferred to the muslin filter. The fiber was then washed once on the filter with cold 1.25% hydrochloric acid, and the washing completed with hot water. The transfer of the fiber and the completion of the determination were conducted exactly as described in the previous method. In length of time required this method is found to be only slightly shorter than the double filtration through muslin. The results obtained are seen to be higher in each case than those obtained by any of the other methods.

The results obtained by Mr. Francis' single filtration method through hardened filter are higher than either of the double filtration methods. For example, one of the single filtration methods gives 1.640%, on Sample No. 1, which is lower than any of the results obtained on this sample with our double filtration method and also lower than one of the results by the regular official method filtering once through linen and once through asbestos. On Sample No. 2 one of the determinations, 11.010, is lower than either of the results given by our double filtration method. These results would seem to indicate that the longer heating, caused by starting with the cold acid and alkali solutions, does not reduce the amount of crude fiber. The other modification in the method, by washing once with 1.25% hydrochloric acid, greatly facilitates the removal of the alkali and the chlorids and since it does not appear to affect the final amount of crude fiber its use is to be recommended.

Per cent of crude fiber obtained.

PROCEDURE	SAMPLE 1	SAMPLE 2	REMARKS
(A) Official method, first filtration through linen and second filtration through asbestos:	1.520 1.525 1.750	10.045 10.140 10.180	8-10 hours, filtration difficult.
Average.....	1.598	10.122	

Per cent of crude fiber obtained.—Continued.

PROCEDURE	SAMPLE 1	SAMPLE 2	REMARKS
(B) Smith-Francis method of filtration through hardened filter on Büchner funnel:	1.640 1.845 1.805	11.160 11.010 11.245	6-7 hours, filtration and transfer difficult.
Average.....	1.763	11.138	
(C) Missouri Station method, 2 filtrations through muslin, starting with cold 1.25% acid and alkali:	1.715 1.780 1.740	Lost 11.105 11.060	About 4½ hours, muslin used 98 x 102 threads per inch.
Average.....	1.745	11.083	
(D) Sweeney method, one filtration through muslin:	1.845 1.845 1.840	11.475 11.215 11.560	3½-4 hours.
Average.....	1.843	11.417	

REPORT ON SUGAR.

By C. A. BROWNE (Sugar Trade Laboratory, New York, N. Y.),
Referee.

Since the report of Mr. Cross in 1913, the work of the Association upon sugar has been interrupted, no referee having been appointed for the year 1913-1914.

In resuming the work which was so efficiently carried out by his predecessor, it occurred to the present referee that this Association during the next few years might in a measure retrieve a part of the damage which has resulted to the progress of sugar analysis as a consequence of the European war. Two international congresses had been scheduled this year to report upon sugar, that of the International Congress of Applied Chemistry, which was to meet in Petrograd in August, and that of the International Commission upon Uniform Methods of Sugar Analysis, which was to meet in Amsterdam in September. Both of these meetings have been indefinitely postponed and many years must elapse before the important questions which they were to consider are reported.

At the last meeting of the International Commission upon Uniform Methods of Sugar Analysis in New York in 1912, a committee was appointed to study the inversion constant of the Clerget method. As the present referee, in connection with his duties upon this committee, is conducting a series of researches along this line, he has selected the pres-

ent occasion as an opportune one in which to report upon certain modifications of the Clerget method.

A STUDY OF CERTAIN MODIFICATIONS OF THE CLERGET METHOD

The volume to which the solution is made up for invert polarization which was formerly used by this Association and which is still employed by many sugar chemists, is the one originally proposed by Clerget. In this procedure 50 cc. of the clarified solution used for direct polarization were transferred to a flask graduated at 50 cc. and 55 cc. Concentrated hydrochloric acid was then added to the 55 cc. mark, the solution was mixed, a thermometer was inserted and the inversion was carried out according to the regulations prescribed for temperature and time of heating. After cooling, the solution was read upon the polariscope in a 200 mm. tube and the observed reading was increased by one-tenth to correct for the dilution from 50-55 cc. This corrected reading and the temperature of the solution were then substituted in the Clerget formula for calculating the percentage of sucrose.

One great advantage of diluting the solution from 50-55 cc. is that any errors in reading the inverted solution are increased by only one-tenth, whereas in the modifications which dilute from 50-100 cc. these errors are multiplied by 2. But notwithstanding this advantage, the experience of chemists has shown a certain unreliableness to exist in the old Clerget procedure so that in later years this method of making up to volume has been largely discontinued.

The referee believes that one great cause of the difficulty, which has been experienced with the old Clerget procedure, has been the neglect of a very serious error, viz: the diminution in volume which takes place in the 55 cc. of solution during inversion. This diminution of volume is due to three causes:

(1) The contraction in volume which all sucrose solutions undergo during inversion and which for 13 grams of sucrose in 55 cc. is about one-fourth of a cubic centimeter.

(2) The elevation in temperature produced by the addition of the hydrochloric acid. This elevation, for 5 cc. of concentrated hydrochloric acid to 50 cc. of sugar solution, is about 3°C., the cooling of the solution from 23°C. at the beginning to 20°C. at the end of the inversion produces a further slight contraction.

(3) The evaporation of water from the neck of the flask during inversion, the amount of such evaporation depending upon the diameter of the neck of the flask, and the time and temperature of inversion.

The combined influence of these 3 factors causes the volume of the 55 cc. of solution at the end of inversion to be about one-third of a cubic centimeter too small for the half normal weight of 13 grams.

It is essential to the accuracy of any method of double polarization that the volume of the solution after inversion be fixed with the utmost accuracy. The sources of error just mentioned are easily overcome if the volume of the inverted solution be brought to 55 cc. at the end of inversion and not at the beginning. If this precaution be followed more accurate results can be secured by the process of making the inverted solution up to 55 cc. than by any of the modifications which dilute to 100 cc. The control of temperature by this procedure is easily carried out by means of a thermometer placed in a control flask containing 55 cc. of blank solution. There is no danger of irregular mixing of the acid with the sugar solution, as the diffusion of the hydrochloric acid through the body of the liquid takes place quickly without shaking.

In employing this method the referee makes all his inversions at room temperature; 5 cc. of concentrated hydrochloric acid were added to the 50 cc. of sugar solution in a 50-55 cc. flask and after standing overnight the volume was completed to exactly 55 cc. at 20°C., after gently tapping the walls of the flask to detach any air bubbles which might have accumulated. The solution is then mixed and read in a polariscope which is also at a temperature of 20°C. The invert reading is then corrected by adding the necessary one-tenth.

The formula for calculating the sucrose by this method of inversion, when a normal weight of 26 grams of pure sucrose is taken, is

$$S = \frac{100 (A - B)}{144.9 - \frac{t}{2}}$$

The above formula, corrected for the differences in specific rotation of invert sugar due to the varying concentration, when less than 26 grams of sucrose are dissolved to 100 cc., is

$$S = \frac{100 (A - B)}{144.9 - \frac{t}{2} - 0.01 \left[144.9 - \frac{t}{2} - (A - B) \right]} \quad \text{in which}$$

S = per cent of sucrose;

A = direct polarization;

B = corrected invert polarization;

t = temperature of the invert solution.

If the invert polarization be made at exactly 20°C. the above formula becomes

$$S = \frac{100 (A - B)}{134.9 - 0.01 [134.9 - (A - B)]}$$

The determinations of Table 1 give an idea of the comparative accuracy of this method when applied to solutions of pure sucrose.

TABLE 1.

Application of Clerget modified procedure to solutions of pure sucrose.

NUMBER	DIRECT POLARIZATION (A)	CORRECTED INVERT POLARIZATION (B)	TEMPERATURE (t)	SUCROSE (S)	
				Found	Taken
			°C.	per cent	per cent
1	99.90	-35.00	19.8	99.92	99.90
2	76.95	-26.79	19.6	76.97	76.92
3	57.70	-19.69	20.2	57.66	57.69
4	38.45	-13.04	20.2	38.44	38.46
5	19.20	- 6.49	20.0	19.15	19.23
6	7.70	- 2.53	20.0	7.65	7.69

TABLE 2.

Application of Clerget modified procedure to solutions of sucrose and dextrose.

NUMBER	DEXTROSE	DIRECT POLARIZATION (A)	CORRECTED INVERT POLARIZATION (B)	TEMPERATURE (t)	SUCROSE (S)	
					Found	Taken
	grams per 100 cc.			°C.	per cent	per cent
1	4.8	91.70	-11.88	20	76.96	76.92
2	4.8	72.45	- 5.06	20	57.70	57.69
3	4.8	53.15	+ 1.60	20	38.45	38.46
4	4.8	33.90	+ 8.20	20	19.23	19.23

The importance of using a formula, which corrects for the differences in specific rotation of invert sugar due to varying concentration, may be illustrated by taking the case of determination No. 3 in Table 2. This determination, using the uncorrected formula, gives 38.21 per cent sucrose, which is a quarter of a per cent too low.

In this connection the referee would very urgently recommend that the Association adopt a modified Herzfeld formula, or the Herzfeld table of factors, in place of the uncorrected formula¹. The correction of the Herzfeld factor 142.66, for concentration has been done in several ways.

According to one method the corrected Herzfeld factor equals $141.78 + 0.0676 C$, C being the grams of sucrose in 100 cc. The objection to this method is that a preliminary calculation with the uncorrected factor 142.66 is necessary in order to obtain an approximate value of C for determining the true factor. The work of calculation is thus doubled.

According to another method the corrected Herzfeld factor equals $141.84 + 0.05 n$, in which n is the scale reading of the inverted solution. The objection to this second method is that it applies to inverted solutions of pure sucrose and is of no value when other optically active substances are present.

¹ U. S. Bur. Chem. Bull. 107, Rev., p. 41.

The two objections named are overcome if the correction for concentration be based upon the difference between the direct and invert polarization. The Herzfeld formula as thus modified becomes

$$S = \frac{100 (A - B)}{142.66 - \frac{t}{2} - 0.0065 \left[142.66 - \frac{t}{2} - (A - B) \right]}$$

which for 20°C. is simplified to

$$S = \frac{100 (A - B)}{132.66 - 0.0065 [132.66 - (A - B)]}$$

The above modified Herzfeld formula is the one which the referee would recommend to the Association for adoption.

A great deal of work has been done by different investigators during the past few years upon methods of double polarization in the effort to make them better adapted to the analysis of sugar mixtures. In the ordinary methods of procedure any levulose, or other sugar, whose optical activity is changed by the addition of the inverting acid, has a different rotation before and after inversion so that a considerable error may be introduced in the calculation of sucrose. The possible magnitude of this error may be seen from the determinations given in Table 3 which were made upon known mixtures of sucrose and invert sugar. The modified procedure of the original Clerget method was used.

TABLE 3.

Application of Clerget modified procedure to solutions of sucrose in invert sugar.

NUM- BER	INVERT SUGAR	DIRECT POLAR- IZATION (A)	CORRECTED INVERT POLAR- IZATION (B)	TEMPERATURE (t)	SUCROSE (S)	
					Found	Taken
	<i>grams per 100 cc.</i>			<i>°C.</i>	<i>per cent</i>	<i>per cent</i>
1	1.10	94.60	-35.20	19.8	96.18	96.15
2	2.74	73.35	-30.42	20.0	77.10	76.92
3	5.48	50.70	-27.28	20.4	58.14	57.69
4	8.21	28.15	-24.04	20.4	38.99	38.46
5	9.86	6.90	-19.58	20.4	19.82	19.23

It is seen that as the amount of invert sugar increases there is a corresponding increase in the sucrose error. In case of honeys and other products containing a high percentage of levulose the error in the sucrose determination due to this cause may exceed 1%.

In mixtures of sucrose with invert sugar alone, it is possible to correct this error in the determination of sucrose by means of an empirical factor. Such a method of correction is not adapted, however, to the analysis of complex mixtures of optically active substances. The efforts of chemists

have been directed, therefore, towards devising a method by which the direct and invert polarizations could be made under similar conditions.

Of the various methods which the referee has tested in this connection, the one which has given the most satisfactory results was that by means of invertase.

The use of invertase from yeast has long been recognized by chemists as the method of inversion which is most perfect in theory, but the difficulty of preparing satisfactory solutions of invertase has been a great obstacle against the introduction of the method. In 1909 Mr. Hudson devised several improvements for preparing invertase from yeast and in April 1910 published a paper upon the use of his invertase solution in the quantitative estimation of sucrose. In Hudson's method 50 cc. of the clarified solution used for the direct polarization are transferred to a 100 cc. flask, faintly acidified with acetic acid and then treated with 5 cc. of stock invertase solution. The volume is completed to 100 cc. and after inversion the solution read in the usual way, the invert reading being corrected for the optical activity of the invertase solution. The formula proposed by Mr. Hudson for this modification of the Clerget process is

$$S = \frac{100 (A - B)}{141.7 - \frac{t}{2}}$$

The assertion has been made that the influence of concentration upon the Clerget factor would practically disappear, provided the inverting agent was without influence upon the rotation of the invert sugar. This, however, is not the case and cannot be the case, since the specific rotation of invert sugar necessarily increases with the concentration. The referee has subjected the invertase method to a most careful examination and finds that the influence of concentration is just as pronounced as with the methods which employ hydrochloric acid.

The invertase solution employed by the referee was prepared according to the original procedure of Mr. Hudson and had the following properties:

Sp. gr. at $\frac{20^{\circ}\text{C}}{20^{\circ}} = 1.00363$;

Refractive index = 1.33468;

Solids in 10 cc. solution = 0.1027 grams;

Ash in 10 cc. solution = 0.0040 grams;

Rotation of solution in 200 mm. tube = $+0.65^{\circ}$ Ventzke.

In using this invertase solution for the Clerget determination the referee followed the method proposed by Mr. Hudson except that the 50 cc. of sugar solution were treated with 10 cc. of invertase reagent in-

stead of 5 cc. and the solution was warmed to 50°C., to hasten the inversion. The solution after standing overnight was made up to 100 cc. at 20°C. and polarized at this temperature. The invert polarization was then increased by 0.0065 to correct for the dextro-rotatory effect of the 10 cc. of invertase solution and then multiplied by 2 to correct for the dilution to 100 cc. The formula for calculating the percentage of sucrose by this method, as calculated from the average of many determinations upon sugar solutions of different concentration, was found to be

$$S = \frac{100 (A - B)}{142 - \frac{t}{2} - 0.0065 \left[142 - \frac{t}{2} - (A - B) \right]}$$

It will be noted that the concentration factor 0.0065 is the same as that found for the Herzfeld method, which follows the same method of diluting the 13 grams of inverted product to 100 cc.

The application of the above formula to the determination of sucrose in various mixtures with invert sugar is given in Table 4.

TABLE 4.
Application of invertase method to solutions of sucrose and invert sugar.

NUM- BER	INVERT SUGAR	DIRECT POLAR- IZATION (A)	CORRECTED INVERT POLAR- IZATION (B)	TEMPERATURE (C)	SUCROSE (S)	
					Found	Taken
	<i>grams per 100 cc.</i>			<i>°C.</i>	<i>per cent</i>	<i>per cent</i>
1	0.00	99.80	-31.83	20	99.72	99.80
2	0.18	95.85	-30.83	20	95.99	95.96
3	0.43	89.75	-29.33	20	90.27	90.20
4	0.67	85.60	-28.23	20	86.31	86.37
5	0.92	79.50	-26.73	20	80.58	80.61
6	2.44	47.00	-18.63	20	49.89	49.90
7	2.44	27.90	-12.43	20	30.69	30.71

The results show that the invertase method gives far more accurate results than the method of acid inversion in the analysis of complex mixtures which contain sucrose and levulose.

The referee does not believe that the invertase method will immediately displace the method of acid inversion for purposes of commercial analysis. The preparation of the invertase solution is somewhat troublesome and the gradual deterioration of the reagent involves the risk of incomplete inversion. The process is one which requires care and watchfulness. A blank analysis upon a weighed amount of pure sucrose should be carried out with every set of determinations. The invertase method is invaluable, however, as a control upon the accuracy of the method of acid inversion and the referee would recommend that it be adopted as a provisional method by the Association.

RECOMMENDATIONS.

In planning the work for next year the referee believes that attention should be devoted to those matters which are of most pressing importance to sugar analysis. He would, therefore, recommend—

(1) That the study of the modifications of the Clerget method be continued with special reference to the accurate determination of sucrose in complex mixtures of carbohydrates.

(2) That efforts be made towards the adoption of an accurate method for determining small quantities of invert sugar in the presence of large amounts of sucrose. In this connection the colorimetric methods for estimating invert sugar should be examined.

(3) That next year's referee in collaboration with the U. S. Bureau of Chemistry establish a table of reduction factors for the more common reducing sugars.

There are a number of improvements and changes, which the progress of sugar analysis, during the past 10 years, requires to be made in the official and provisional methods of the Association. The referee has gone over these methods with several of the members of the Committee on Editing Methods of Analysis and it was considered advisable to present the following recommendations:

(4) That the official text of the methods of the International Commission be substituted for the present text¹ and that these methods of the International Commission be brought up to date.

(5) That the following method for preparing a salt-free alumina cream be adopted in place of the present method²:

Precipitate concentrated alum solution with a slight excess of ammonium hydroxid and wash the precipitate by decantation with water until the solution is free from sulphates. Pour off the excess of water and store the residual cream in a stoppered bottle.

(6) That at the end of section (c)³ the following be inserted:

For concentrations of sucrose of less than 13 grams to 100 cc. of invert solution the following general formula should be used.

$$S = \frac{100 (P - I)}{142.66 - \frac{t}{2} - 0.0065 \left[142.66 - \frac{t}{2} - (P - I) \right]}$$

The above formula is applicable to the determination of sucrose in the presence of dextrose, commercial glucose, and all other substances whose optical activity is not affected by the inverting acid. With materials which contain much levulose, such as honey, fruit products, etc., the method gives too high results.

¹ U. S. Bur. Chem. Bull. 107, Rev., pp. 39-40.

² Ibid., p. 40.

³ Ibid., p. 41.

(7) That at the end of section (d)¹ the following formula be substituted for the present formula for calculating the per cent of sucrose in the presence of raffinose:

$$S = \frac{0.5124 P - I}{0.839}$$

The above formula supposes that the polarizations be made at exactly 20°C. If the temperature (T) be other than 20°C., the following formula should be used:

$$S = \frac{P (0.4724 + 0.002 T) - I}{0.899 - 0.003 T}$$

Having calculated S,

$$R = \frac{P - S}{1.852}$$

(8) That after section (d)¹ the following be inserted:

DETERMINATION OF SUCROSE BY MEANS OF INVERTASE.—PROVISIONAL.

Preparation of Invertase Solution, Hudson Method.

Break up 5 pounds of pressed yeast, which may be either baker's or brewer's yeast, add 30 cc. of chloroform to it in a closed flask and allow to stand at room temperature (20°C.) overnight. By morning the solid mass will have become fluid and it should then be filtered through filter paper allowing several hours for draining. To the filtrate add neutral lead acetate until no further precipitate forms and again filter. Precipitate the excess of lead from the filtrate with potassium oxalate and filter. To this filtrate add 25 cc. of toluene and dialyze the mixture in a pig's bladder for 2 or 3 days against clear running tap water. The dialyzed solution should be colorless, perfectly clear after filtration, and neutral to litmus; it should be preserved in an ice-box with the addition of a little toluene to prevent the growth of micro-organisms. The optical activity of the invertase solution is noted and a correction for this, according to the amount of solution used, must be applied to the invert reading.

Determination.

Dissolve the normal weight (26 grams) of substance in water, clarify, make up to volume, and take the direct polarization (P) as under section (d). Remove the excess of lead from the filtrate, if lead has been used as a clarifying agent, with anhydrous sodium carbonate or potassium oxalate, and filter. To 50 cc. of the filtrate in a 100 cc. flask add acetic acid by drops until the reaction is acid to litmus, add 10 cc. of the stock invertase solution and let stand in a warm place (about 40°C.) overnight. Cool and make up to 100 cc. at 20°C. Polarize at 20°C. in a 200 mm. tube. Allow the solution to remain in the tube for an hour and repeat the polarization. If there is no change from the previous reading the inversion is complete, when the reading and temperature of the solution are carefully noted. The reading is corrected for the optical activity of the invertase solution and then multiplied by 2. The percentage of sucrose is then calculated by the following formula:

¹ U. S. Bur. Chem. Bull. 107, Rev., p. 41.

$$S = \frac{100 (P - I)}{142 - \frac{t}{2} - 0.0065 \left[142 - \frac{t}{2} - (P - I) \right]}$$

S = per cent of sucrose;

P = direct reading;

I = invert reading;

t = temperature at which invert reading is made.

(9) That before section (c)¹ the following be inserted:

Reducing sugars other than dextrose may be determined, using Allihn's modification of Fehling's solution, by means of the above table and method by use of the following factors:

Arabinose	= Glucose \times 0.969;
Xylose	= Glucose \times 1.017;
Levulose	= Glucose \times 1.093;
Invert sugar	= Glucose \times 1.044;
Galactose	= Glucose \times 1.114.

(10) That before section (3. Ash²) the table for specific gravity and total solids of sucrose solutions at $\frac{20^{\circ}\text{C.}}{4}$ used by the U. S. Bureau of Standards and Reichsanstalt of Germany be inserted.

(11) That in the Munson and Walker table³ for calculating lactose the column for lactose with one-half molecule of water be omitted.

No report was made by the referee on the general subject of the separation of nitrogenous substances.

REPORT ON THE SEPARATION OF NITROGENOUS SUBSTANCES (MEAT PRODUCTS).

By P. F. TROWBRIDGE (Agricultural Experiment Station, Columbia, Mo.), *Associate Referee*.

No collaborative work has been undertaken. In connection with other work, a number of carefully prepared samples have been secured upon which it is planned to study the products of dissociation.

It is recommended that the referee for next year attempt to determine the relative amounts of some of the dissociation products in water-soluble and water-insoluble meat proteins.

¹ U. S. Bur. Chem. Bull. 107, Rev., p. 51.

² Ibid., p. 67.

³ Ibid., pp. 243-51.

REPORT ON THE SEPARATION OF NITROGENOUS SUBSTANCES (MILK AND CHEESE).

By LEROY S. PALMER (Agricultural Experiment Station, Columbia, Mo.),
Associate Referee.

The recommendation adopted for this work at the last meeting of the Association called for studies leading to the adoption of methods for the determination of the non-casein proteins and the products of protein decomposition in milk. Your referee begs to report that a serious illness last spring prevented his giving this work the attention originally planned. Some progress has been made, however, and although the results secured to date are too indefinite for detailed report, the work is being continued.

In the report made last year attention was called to the fact that the combined official method of the Association for casein, and the provisional method for the so-called albumin does not remove all the substances from milk that are of true protein character. This is particularly true in old milk that has undergone protein decomposition; but the writer wishes to point out that even the very freshest milk will usually show a non-casein-non-heat-coagulable-protein percentage approximately equal to the percentage of heat-coagulable protein obtained by the present provisional method. The following table shows the analyses of six 10 cc. portions of the same skim milk¹, the analyses being started about 3 hours after the milk was drawn.

Per cent of protein in milk by different methods.

METHOD	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 4	SAMPLE 5	SAMPLE 6
Total protein (official).....	3.80	3.80	3.80	3.80	3.80	3.80
Casein (official).....	2.89	2.89	2.89	2.84	2.84	2.88
Heat-coagulable proteins (provisional)....	0.526	0.502	0.451	0.529	0.544	0.510
Residual proteins (precipitated by Almén's reagent).....	0.280	0.280	0.288	0.273	0.322	0.274
Residual nitrogen (as protein not precipitated by Almén's reagent).....	0.160	0.205	0.301	0.232	Lost	0.205

Other figures could be presented showing relatively the same results for other samples of milk. The preceding figures show approximately 8% of the total true protein of fresh milk not recovered by acid precipitation or heat coagulation, a percentage sufficiently great to demand attention. Unless further study shows that these residual proteins, at least those precipitated by Almén's tannic acid reagent, are formed as the result of the present methods of analysis for casein and heat-coagulable protein, it would seem that the presence of these simpler protein substances in fresh milk should be recognized, and suitable methods incorporated in

¹ Mixed milk from the University herd.

the official methods for their determination. Careful distinction should be made at the same time between these substances present in fresh milk, and the products of protein decomposition, some of which would be determined by the same method. The data at least serve to call attention to the need of a careful study of the origin and exact character of these protein bodies present in fresh milk, which would ordinarily be called proteoses and peptones.

RECOMMENDATION.

It is recommended—

(1) That the studies be continued leading to the adoption of methods for the determination of the non-casein proteins and the products of protein decomposition in milk.

REPORT ON DAIRY PRODUCTS¹.

By LEWIS I. NURENBERG (State Department of Health, Boston, Mass.)

Referee.

A study, and compilation of figures, has been made of the sour serum method for the detection of added water in milk. In this work the referee has had the coöperation of Messrs. J. T. Keister, associate referee, and L. W. Ferris, of the Bureau of Chemistry, Washington, D. C., and George B. Taylor, J. R. Keeney, and J. S. Slack, of the State Board of Health, New Orleans, La.

The referee has obtained figures of over 660 samples of known purity milk from individual cows, and 49 samples from herds. These samples are representative of practically all breeds of cows mentioned in the 1914 report and of such influencing conditions as variation in season and lactation period. More than one-half of the total number represent low-grade milk.

The methods of preparation and analysis of the serum are as follows:

PREPARATION OF THE SOUR SERUM².

Allow the milk to sour spontaneously, and filter.

REFRACTION OF SOUR SERUM.

Determine the index of refraction of the clear serum at 20°C. by means of the Zeiss immersion refractometer. A refraction below 38.3 indicates added water.

Transfer 25 cc. of the serum to a flat-bottomed platinum dish and evaporate to dryness over the water bath. Then heat the contents of the dish over the small flame (to avoid sputtering) until charred. Place the dish in an electric muffle, with pyrometer connected, and ash at a heat not greater than 500°C., or 900°F. Cool and weigh. Express result in grams per 100 cc. An ash below 0.730 indicates added water.

¹ Presented by H. C. Lythgoe.

² Matthes and Müller. *Z. offic. Chem.*, 1903, 9: 173-8.

In the original article as described by A. Burr and F. M. Berberich¹, the authors have outlined the following method:

ASH OF SOUR SERUM.

Measure 50 cc. of the serum into a platinum dish, evaporate to dryness, and carbonize over a low flame. Extract the char with hot water, burn the insoluble residue, add the solution to this ash, evaporate to dryness, ignite at a low temperature and weigh.

Experience has shown, however, with the exercise of care and an accurate pyrometer that the former stated method is as accurate as the latter, and involves less manipulation.

Table 1 contains the refractions of the sour serum of 660 samples of known purity milk from individual cows and 49 samples from herds. In the case of the individual cows the variation in the readings extends from 38.3–45.9. The greatest percentage of samples (25.2%) refracts between 41 and 42. There appears to be a certain uniformity each side of this medial point. The percentage of samples refracting between 40 and 41, and 42 and 43 is practically the same (from 18–20%); between 39 and 40, and 43 and 44 the percentage is from 12–13.5%, while from 38.3–39 and 44–45 the percentage is 4–5%.

In the herds the largest percentage of samples (75%) refracts between 41 and 43. Between 40 and 41 and 43 and 44 the percentage is from 10–12. As would be expected, no samples were found refracting at the maximum (45.9) or minimum (38.3) limits.

TABLE 1.
Variation in refractive indices of sour serum of known purity milk.
(660 samples from individual cows; 49 samples from herds.)

INDIVIDUAL COWS			HERDS		
Reading at 20°C.	Number of samples examined	Per cent of samples examined	Reading at 20°C.	Number of samples examined	Per cent of samples examined
38.3–38.9	34	5.2	38.3–38.9	0	0
39–39.9	81	12.3	39–39.9	2	4.1
40–40.9	121	18.3	40–40.9	6	12.3
41–41.9	167	25.2	41–41.9	18	36.7
42–42.9	133	20.2	42–42.9	18	36.7
43–43.9	90	13.6	43–43.9	5	10.2
44–44.9	27	4.1	44–44.9	0	0
45–45.9	7	1.1	45–45.9	0	0

Following is the graphic representation of the figures given in Table 1. The curves cross at the 20% line with a refractive index of 40.8 and again at the 15% line with a refractive index of 43.3.

¹ Chem. Ztg., 1908, 32: 617–8.

VARIATION IN REFRACTIVE INDICES OF SOUR SERUM OF KNOWN PURITY MILK.

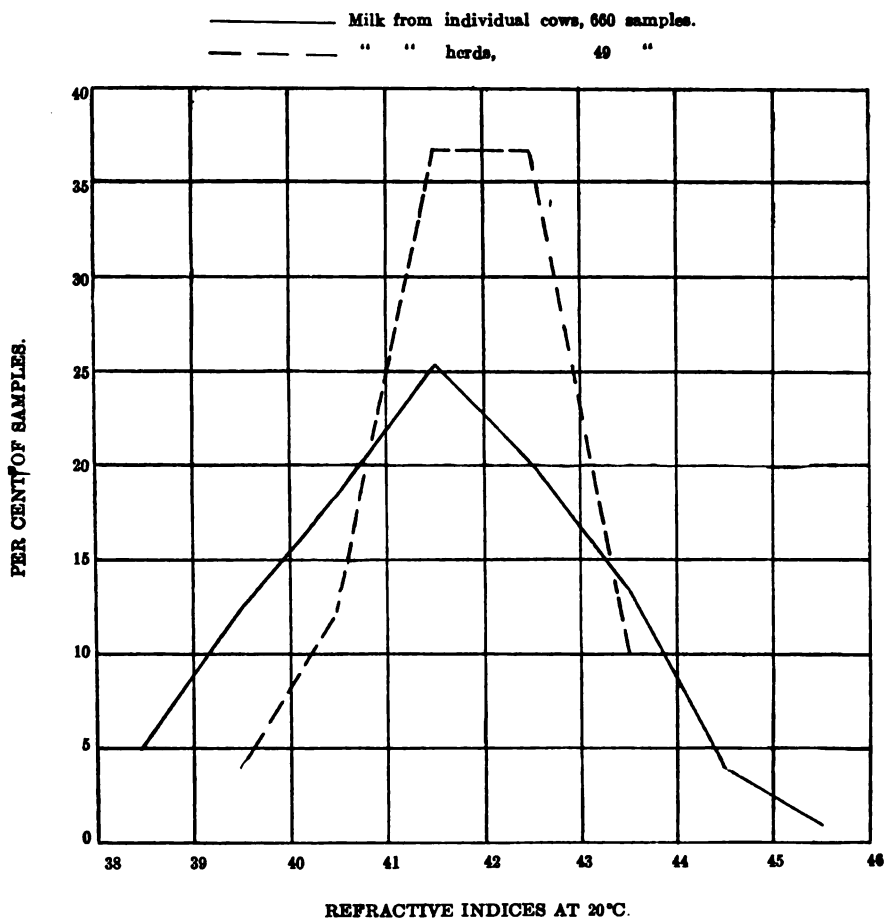


TABLE 2.

Variation in ash of sour serum of known purity milk.
(504 samples from individual cows; 35 samples from herds.)

INDIVIDUAL COWS			INDIVIDUAL COWS			HERDS		
Ash gram per 100 cc.	Number of samples examined	Per cent of samples examined	Ash gram per 100 cc.	Number of samples examined	Per cent of samples examined	Ash gram per 100 cc.	Number of samples examined	Per cent of samples examined
0.730-0.739	41	8.2	0.840-0.849	17	0.730-0.739
0.740-0.749	54	0.850-0.859	7	4.8	0.740-0.749	1	2.9
0.750-0.759	34	17.3	0.860-0.869	16	0.750-0.759	2
0.760-0.769	51	0.870-0.879	7	4.6	0.760-0.769	4	17.1
0.770-0.779	38	17.7	0.880-0.889	7	0.770-0.779	2
0.780-0.789	52	0.890-0.899	4	2.2	0.780-0.789	6	22.8
0.790-0.799	45	19.3	0.900-0.909	4	0.790-0.799	6
0.800-0.809	51	0.910-0.919	0.8	0.800-0.809	9	42.9
0.810-0.819	37	17.3	0.920-0.929	1	0.810-0.819	4
0.820-0.829	26	0.930-0.939	1	0.4	0.820-0.829	1	14.3
0.830-0.839	9	7.0	0.940-0.949	2	0.4	0.830-0.949

Table 2 presents the sour serum ash figures of 504 samples of known purity milk from individual cows and 35 samples from herds. In the individual cows only 8.2% of the ash figures extended between the minimum limit 0.730 and 0.740, while 71.6% were between 0.740 and 0.820; 16.4% of the figures were between 0.820-0.880, and 3.8% between 0.880-0.950. In the milk from herds no samples were found with ash figures from the minimum limit 0.730-0.739, or from 0.830 to the maximum limit 0.949; 2.9% of the ash figures were between 0.740-0.749; 82.8% were between 0.750 and 0.810; and 14.3% were between 0.810 and 0.830.

The data from Table 2 are shown in the following plot. The curves cross at the 17.5% line with an ash percentage of 0.761.

VARIATION IN ASH OF SOUR SERUM OF KNOWN PURITY MILK.

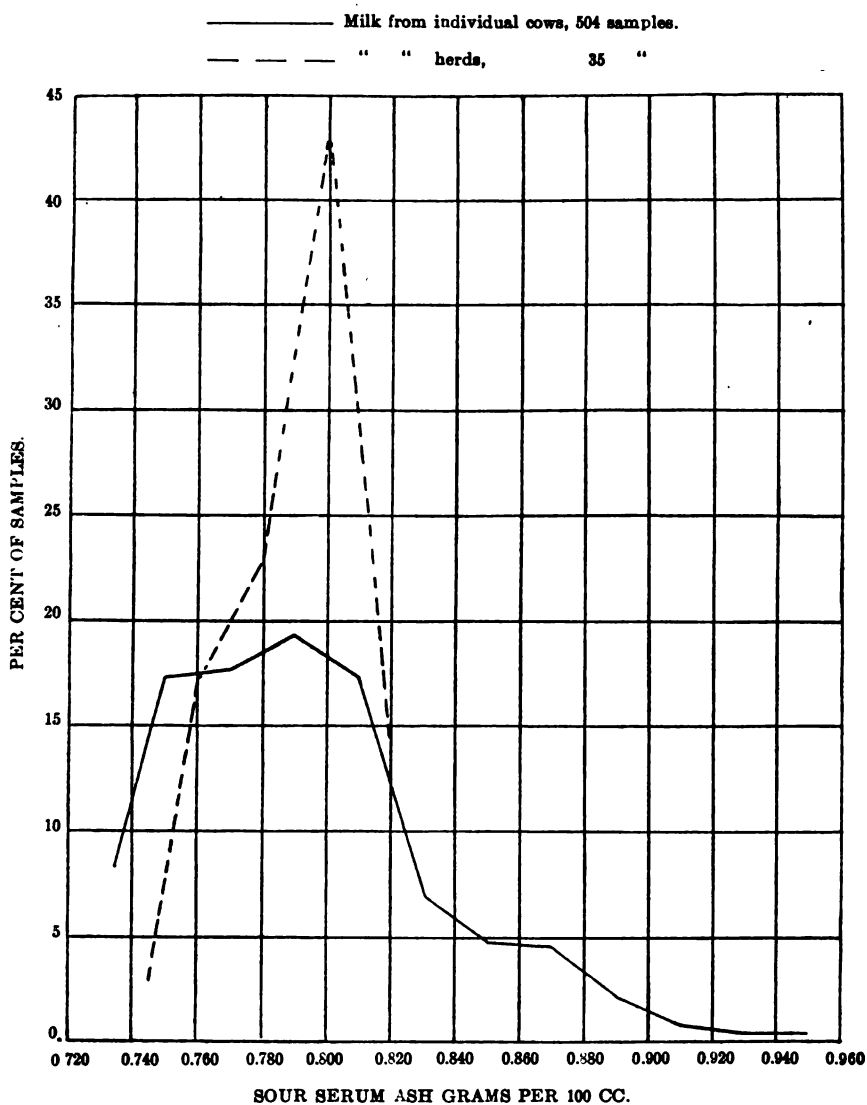


TABLE 3.

Variation in refractive indices and ash of sour serum of known purity milk.
From individual cows.

REFRACTIVE INDICES	NUMBER OF SAMPLES EXAMINED	PER CENT OF SAMPLES EXAMINED	ASH GRAM PER 100 CC.	NUMBER OF SAMPLES EXAMINED	PER CENT OF SAMPLES EXAMINED	ASH GRAM PER 100 CC.	NUMBER OF SAMPLES EXAMINED	PER CENT OF SAMPLES EXAMINED
38.0-38.4	20	3.0	0.730-0.734	30	5.9	0.840-0.844	8	1.6
38.5-38.9	14	2.1	0.735-0.739	11	2.2	0.845-0.849	9	1.8
39.0-39.4	45	6.8	0.740-0.744	33	6.5	0.850-0.854	3	0.6
39.5-39.9	36	5.5	0.745-0.749	21	4.1	0.855-0.859	4	0.8
40.0-40.4	60	9.1	0.750-0.754	19	3.8	0.860-0.864	8	1.6
40.5-40.9	61	9.2	0.755-0.759	15	3.0	0.865-0.869	8	1.6
41.0-41.4	87	13.2	0.760-0.764	33	6.5	0.870-0.874	5	1.0
41.5-41.9	80	12.1	0.765-0.769	18	3.6	0.875-0.879	2	0.4
42.0-42.4	87	13.2	0.770-0.774	26	5.2	0.880-0.884	4	0.8
42.5-42.9	46	7.0	0.775-0.779	12	2.4	0.885-0.889	3	0.6
43.0-43.4	52	7.9	0.780-0.784	31	6.1	0.890-0.894	1	0.2
43.5-43.9	38	5.8	0.785-0.789	21	4.1	0.895-0.899	3	0.6
44.0-44.4	18	2.7	0.790-0.794	25	4.9	0.900-0.904	3	0.6
44.5-44.9	9	1.4	0.795-0.799	20	4.0	0.905-0.909	1	0.2
45.0-45.4	5	0.7	0.800-0.804	32	6.3	0.910-0.914
45.5-45.9	2	0.3	0.805-0.809	19	3.8	0.915-0.919
Total.....	660	100.0	0.810-0.814	27	5.4	0.920-0.924	1	0.2
			0.815-0.819	10	2.0	0.925-0.929
			0.820-0.824	20	4.0	0.930-0.934	1	0.2
			0.825-0.829	6	1.2	0.935-0.939
			0.830-0.834	7	1.4	0.940-0.944	2	0.4
			0.835-0.839	2	0.4	Total.....	504	100.0

From herds.

REFRACTIVE INDICES	NUMBER OF SAMPLES EXAMINED	PER CENT OF SAMPLES EXAMINED	ASH GRAM PER 100 CC.	NUMBER OF SAMPLES EXAMINED	PER CENT OF SAMPLES EXAMINED
38.0-38.4	0.730-0.734
38.5-38.9	0.735-0.739
39.0-39.4	0.740-0.744	1	2.9
39.5-39.9	2	4.1	0.750-0.754	2	5.7
40.0-40.4	4	8.2	0.760-0.764	2	5.7
40.5-40.9	2	4.1	0.765-0.769	2	5.7
41.0-41.4	8	16.3	0.770-0.774	1	2.9
41.5-41.9	10	20.4	0.775-0.779	1	2.9
42.0-42.4	11	22.4	0.780-0.784	2	5.7
42.5-42.9	7	14.3	0.785-0.789	4	11.4
43.0-43.4	4	8.2	0.790-0.794	4	11.4
43.5-43.9	1	2.0	0.795-0.799	2	5.7
Total.....	49	100.0	0.800-0.804	4	11.4
			0.805-0.809	5	14.3
			0.810-0.814	4	11.4
			0.820-0.824	1	2.9
			Total.....	35	100.0

it can be said that sufficiently representative samples have been taken to draw definite conclusions.

The medial line (50% line) shows that 50% of the samples had refractive indices below 41.4 from individual cows, and 41.8 from herds. The curves for the refractive indices of the herds and individual cows cross at the 73% point with refractive index of 42.4. The medial line also shows that in 50% of the samples the sour serum ash was below 0.784 from individual cows and 0.793 from herds. The curves for the sour serum ash of the herds and individual cows cross at the 68% line with the sour serum ash of 0.802.

Table 4 contains the results submitted by Mr. L. W. Ferris of the U. S. Bureau of Chemistry. His experimental work has shown conclusively that the time of spontaneous souring affects the sour serum ash. In the cases of partial souring the sour serum ash figure is lower than the acetic serum ash which, of course, should not be. Unless the sample is sufficiently sour, some of the calcium salt is precipitated with the casein and the sour serum ash is very liable to be low, as shown in Table 4. The minimum sour serum ash figure, reported as 0.671, on longer souring was raised to 0.722 which corresponds to the figure obtained by the acetic serum after being multiplied by 1.02.

COMMENTS BY ANALYSTS.

All samples except the first 8 represent single milkings from individual cows.

The acetic acid serum was obtained by adding 2 cc. of 25% acetic acid to 100 cc. of milk. The results were multiplied by 1.02 to make them comparable with the results on the spontaneous serum. The results obtained by both methods are about the same. The degree of souring of the spontaneous serum appears to make no difference in either the ash or refraction, although clearer serums were obtained by allowing the sample to stand until the whey and curd had separated. In some cases it was impossible to get a clear serum. The spontaneous souring involves less work than the acetic acid method and in most cases the spontaneous serum is about as clear as that obtained by the acetic acid method.

One sample gave a refraction of 37.9 (cow No. 15) on the spontaneous serum and samples from 4 different cows (Nos. 2, 5, 6 and 7) gave an ash of serum below 0.73.

Table 5 contains the results submitted by Mr. J. T. Keister, associate referee. In this table there are 3 abnormal sour serum ash figures. The referee is of the opinion that in each case the milk was not thoroughly sour. If the sample is thoroughly sour, a clear serum will be obtained which will contain all the mineral matter.

TABLE 4.
Data for detection of added water to milk. (September-October, 1916.)
(Leslie W. Ferris, Washington, D. C.)

LABO- RATORY NUM- BER	NUM- BER OF COW	BREED	AGE OF COW	TIME SINCE CALVING	AMOUNT OF MILK	TIME OF MILKING	SPONTANEOUS SERUM			ACETIC ACID SERUM		
							Refraction at 20°C.	Ash	Lactic acid	Refraction at 20°C.	Ash X 1.02	Lactic acid
			years	months	pounds		reading	gram per 100 cc.	gram per 100 cc.	reading	gram per 100 cc.	gram per 100 cc.
9420½		Market milk					41.6	0.798		42.3	0.788	0.727
9421		Market milk					42.0	0.766		42.3	0.796	0.691
9422		Market milk					41.9	0.773				
		Same sample, longer souring					41.5	0.785	0.596			
9423		Market milk					40.7	0.782	0.508	42.0	0.780	0.657
		Same sample, longer souring					41.1	0.781	0.601			
9423		Market milk					41.5	0.775	0.443	41.7	0.783	0.695
		Same sample, longer souring					40.7	0.776	0.508			
9428	1	Holstein	4½	6	20	1 p.m.	39.7	0.802	0.639	40.2	0.804	0.634
		Same sample, longer souring					39.9	0.802	0.675			
9434	1	Holstein	4½	6	20	1 p.m.	39.7	0.801	0.497	39.4	0.814	0.733
		Same sample, longer souring					39.9	0.802	0.675			
		Same sample, longer souring					39.4	0.803	0.695			
9436	1	Holstein	4½	6	12.5	7.30 p.m.	38.7	0.796	0.727	39.7	0.801	0.790
		Same sample, longer souring					39.1	0.811	0.713			
9438	1	Holstein	4½	6	18	4.45 a.m.	39.7	0.812	0.675	40.2	0.815	0.760
9429	2	Holstein	2½	5	19.2	1 p.m.	Cloudy¹	0.717	0.581	41.8	0.722	0.733
		Same sample, longer souring					41.0	0.717	0.648			

TABLE 4.—Continued.

LABO- RATORY NUM- BER OF COW BRE	NUM- BER OF COW	BREED	AGE OF COW	TIME SINCE CALVING	AMOUNT OF MILK	TIME OF MILKING	SPONTANEOUS SERUM			ACETIC ACID SERUM		
							Refraction at 20°C.	Ash	Lactic acid	Refraction at 20°C.	Ash × 1.02	Lactic acid
				<i>months</i>	<i>pounds</i>		<i>reading</i>	<i>gram per 100 cc.</i>	<i>gram per 100 cc.</i>	<i>reading</i>	<i>gram per 100 cc.</i>	<i>gram per 100 cc.</i>
9435	2	Holstein Same sample, longer souring	2½	5	19.2	1 p.m.	40.7	0.709	0.497	41.2	0.709	0.779
		Same sample, longer souring					40.7	0.711	0.544			
9437	2	Holstein Same sample, longer souring	2½	5	12.8	7:30 p.m.	40.7	0.702	0.596			
		Same sample, longer souring					40.7	0.671	0.554	40.7	0.722	0.767
		Same sample, longer souring					40.0	0.687	0.653			
		Same sample, longer souring					40.2	0.722	0.713			
9439	2	Holstein	2½	5	19.0	4:45 a.m.	41.7	0.699	0.544	42.0	0.717	0.751
9448	3	Holstein	5	3	20.7	p.m.	40.7	0.757	0.648	41.2	0.760	
9453	3	Holstein	5	3	23.2	a.m.	41.2	0.757	0.770	41.6	0.752	
9450	4	Holstein	9	9	11.8	p.m.	41.4	0.745	0.563	42.0	0.746	
9454	4	Holstein	9	9	16.8	a.m.	40.7	0.737	0.648	41.7	0.736	
9475	4	Holstein	9	9	13.0	p.m.	40.1	0.732	0.657	41.1	0.734	
9449	5	Holstein	5	4	21.5	p.m.	41.3	0.724	0.535	41.7	0.714	
9456	5	Holstein	5	4	27.5	a.m.	40.9	0.755	0.713	41.5	0.740	
9457	6	Holstein	6	7	12.3	p.m.	42.5	0.739	0.619	41.2	0.773	
9458	6	Holstein	6	7	19.6	a.m.	39.4	0.721	0.554	40.1	0.734	
9474	6	Holstein	6	7	15.5	p.m.	Cloudy	0.709	0.488	39.8	0.716	
9461	7	Holstein	6	8	8.6	p.m.	41.0	0.769	0.555	41.9	0.767	
9462	7	Holstein	6	8		a.m.	41.5	0.712	0.666	40.5	0.726	
9476	8	Holstein	4	2	20.0	p.m.				40.3	0.733	
9443	9	Grade Holstein	8-9	Nearly dry	6.5	p.m.	40.7	0.813	0.742	40.7	0.830	
9444	9	Grade Holstein	8-9	Nearly dry	6.5	a.m.	40.7	0.797	0.665	40.9	0.782	
9452	10	Grade Holstein		3	13½	a.m.	41.7	0.729	0.657	41.8	0.741	
9447	10	Grade Holstein		3	13½	p.m.	41.3	0.732	0.653	41.3	0.735	

9446	11	Grade Holstein	8-9	10	4.3	a.m.	42.7	0.807	0.675	42.7	0.811
9451	12	Grade Holstein	6	10	16.8	a.m.	38.5	0.794	0.517	38.7	0.796
9455	12	Grade Holstein	6	10	17.7	p.m.	41.4	0.816	0.734	39.2	0.818
9459	13	Grade Holstein	8	5.4	p.m.	41.4	0.924*	0.657	Cloudy ⁴	0.877
9460	13	Grade Holstein	8	8.0	p.m.	42.6	0.862	0.716	43.7	0.864
9464	14	Grade Holstein	6	4	19.0	a.m.	40.3	0.822	0.733	42.7	0.816
9465	15	Grade Holstein	9	4	7.0	p.m.	39.1	0.776	0.619	38.0	0.775
9466	15	Grade Holstein	9	4	10.0	a.m.	39.2	0.797	0.657	38.8	0.783
9472	15	Grade Holstein	9	4	7.8	p.m.	37.9	0.766	0.517	37.8	0.769
9467	16	Grade Holstein	2	11	6	p.m.	39.9	0.744	0.601	40.5	0.741
9468	16	Grade Holstein	2	11	8.2	a.m.	40.0	0.767	0.657	40.0	0.745
9473	16	Grade Holstein	2	11	7.4	p.m.	40.5	0.745	0.657	40.9	0.753
9441	17	Durham	5	2	13.0	p.m.	40.7	0.774	0.742	41.2	0.775
9442	18	Durham	2½	1	p.m.	41.0	0.751	0.704	41.3	0.756
9470	19	Durham	6-7	4	17.4	p.m.	41.9	0.756	0.751	42.5	0.736
9471	20	Durham	5	4	17.4	p.m.	41.5	0.803	0.704	41.8	0.785
9440	21	Black Polled	2½	4	13.0	p.m.	39.7	0.763	0.666	40.1	0.786
9445	22	Black Polled	2	4	13.0	a.m.	41.7	0.762	0.733	42.2	0.814
9469	23	Grade Guernsey	3	1	13.0	p.m.	41.4	0.772	0.779	42.1	0.755
Minimum.....											
Maximum.....											
Mean.....											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											
											

TABLE 5.
Analysis of milks from individual cows. (September–October, 1915.)
 (J. T. Keister, Washington, D. C.)

FEDERAL LABORATORY NUMBER	NUM- BER OF COW	BREED	AGE OF COW	TIME SINCE CALVING	AMOUNT OF MILK	TIME OF MILKING	FAT (ROSE- GOTTIERS METHOD)	FAT (BABCOCK METHOD)	SOLIDS NOT FAT (GRAVI- METRIC- ALLY)	SOLIDS NOT FAT (CALCU- LATED)	SOUR SERUM		AGE
											Refraction at 20° C.	Ash	
			years	months	pounds		per cent	per cent	per cent	per cent	reading	gram per 100 cc.	per cent
9434	1 ¹	Holstein	4½	6	15.2	1 p.m.	3.82	3.85	7.96	7.86	39.2	0.748	0.75
9436	1	Holstein	4½	6	12.5	7.30 p.m.	3.71	3.80	7.78	7.78	39.0	0.790	0.76
9438	1	Holstein	4½	6	18.0	4.45 a.m.	2.79	2.80	8.09	8.00	40.0	0.773	0.74
9435	2 ¹	Holstein	2½	5	16.4	1 p.m.	4.22	4.30	8.18	8.12	40.9	0.678	0.657
9437	2	Holstein	2½	5	12.8	7.30 p.m.	3.88	3.95	8.12	8.00	40.3	0.968	0.66
9439	2	Holstein	2½	5	19.0	4.50 a.m.	2.55	2.70	8.52	8.35	40.8	0.704	0.669
9448	3	Holstein	5	3	20.7	p.m.	3.02	3.10	8.33	8.48	40.9	0.753	0.73
9453	3	Holstein	5	3	23.2	a.m.	3.01	3.10	8.44	8.33	41.3	0.764	0.72
9450	4	Holstein	9	9	11.8	p.m.	3.14	3.15	8.74	8.53	41.1	0.754	0.73
9454	4	Holstein	9	9	16.8	a.m.	2.48	2.50	8.76	8.50	40.8	0.756	0.71
9475	4	Holstein	9	9	13.0	3.40 p.m.	2.80	2.80	8.78	8.46	41.1	0.745	0.68
9449	5	Holstein	5	4	21.5	p.m.	3.07	3.15	8.35	8.41	40.4	0.769	0.73
9456	5	Holstein	5	4	27.5	a.m.	3.02	3.15	8.36	8.45	40.9	0.743	0.70
9457	6	Holstein	6	7	12.3	p.m.	3.77	3.90	8.64	8.40	41.3	0.787	0.706
9458	6	Holstein	6	7	19.6	a.m.	3.30	3.05	8.13	7.96	39.2	0.740	0.68
9474	6	Holstein	6	7	15.5	p.m.	4.20	8.05	38.0	0.731	0.664
9461	7	Holstein	6	8	8.6	p.m.	3.31	3.30	8.76	8.56	41.9	0.770	0.69
9462	7	Holstein	6	8	10.0	a.m.	3.21	3.30	8.53	8.31	40.4	0.731	0.74
9476	8	Holstein	4	2	20.0	p.m.	3.75	8.10	39.7	0.729	0.71
9443	9	Grade Holstein	8 or 9	8 or 9	8.7	p.m.	3.47	3.20	8.56	40.9	0.853	0.76
9444	9	Grade Holstein	8 or 9	8 or 9	8.7	a.m.	3.72	3.50	8.63	0.73
9447	10	Grade Holstein	3	3	17.4	p.m.	4.00	8.26	40.4	0.731
9452	10	Grade Holstein	3	17.4	a.m.	3.60	8.59	42.7	0.739
9446	11	Grade Holstein	8 or 9	8 or 10	4.3	a.m.	7.70	8.99	43.2	0.808

9451	12	Grade Holstein	6	10	16.8	p.m.	3.40	3.45	7.49	7.54	41.8	0.801	0.75
9455	12	Grade Holstein	6	10	17.7	a.m.	2.84	2.95	7.65	7.71	41.7	0.813	0.73
9459	13	Grade Holstein	8	5.4	p.m.	4.80	9.30	41.8	0.882
9460	13	Grade Holstein	8	8.0	a.m.	4.40	9.08	42.6	0.898
9464	14	Grade Holstein	6	3	19.0	a.m.	3.10	8.97	41.1	0.832
9465 ¹	15	Grade Holstein	9	4	7.0	p.m.	(1.10) ²	(1.10)	7.48	7.37	40.2	0.781	0.68
9466	15	Grade Holstein	9	4	10.0	a.m.	3.81	3.80	7.79	7.58	40.9	0.806	0.70
9472	15	Grade Holstein	9	4	7.8	p.m.	3.09	3.10	7.60	7.51	38.4	0.790	0.72
9467	16	Grade Holstein	2	11	6.0	p.m.	4.20	8.09	40.0	0.760
9468	16	Grade Holstein	2	11	8.2	a.m.	3.54	3.45	8.22	8.21	41.4	0.754	0.68
9473	16	Grade Holstein	2	11	7.4	p.m.	3.37	3.45	8.47	8.36	41.1	0.770	0.686
9441	17	Grade Durham	5	2	13.0	p.m.	3.75	8.46	40.8	0.782
9442	18	Grade Durham	2½	1	10.9	p.m.	3.50	8.55	41.0	0.745
9470	18	Grade Durham	6	3	17.4	p.m.	3.65	8.98	41.8	0.811
9471	20	Grade Durham	5	3	17.4	p.m.	3.80	8.55	41.8	0.761
9440	21	Black Polled	2½	2	13.0	p.m.	3.18	3.25	8.35	8.26	40.4	0.791	0.767
9445	21	Black Polled	2½	2	13.0	a.m.	3.10	3.20	8.86	8.68	41.9	0.814	0.79
9469	22	Grade Guernsey	3	1	13.0	p.m.	3.85	8.68	41.1	0.779
9478	23	Grade Jersey	20(about)	3	8.7	p.m.	2.83	2.80	8.33	40.4	0.806
9479	24	Grade Jersey	10	1	13.0	p.m.	4.80	7.81	40.1	0.816	0.75
9480	25	Grade Durham	5	1	10.9	p.m.	6.60 ³	8.57	40.6	0.869
9481	26	Grade Durham	10(about)	1	10.9	p.m.	7.90 ⁴	8.85	43.0	0.832
9482 ⁴	Herd	p.m.	4.60	8.42	40.7	0.790

¹ Cows milked 3 times a day.² Sample taken by milker, probably represents first portion of the milking.³ Only 8 hours since previous milking, which probably partly accounts for high fat figure.⁴ Herd test of 10 cows (Nos. 13, 17, 18, 19, 20, 21, 23, 24, 25, 26).

TABLE 6.

Composition of milk from Fagot Dairy, Metairie Ridge, Jefferson Parish.
(G. B. Taylor, J. R. Reeny and J. S. Slack, Louisiana.)

NAME	AGE	BREED	AMOUNT OF MILK	TIME SINCE CALV- ING	SP. GR. AT 15° C.	TOTAL SOLIDS ¹	FAT	SOUR SERUM	
								Ash	Refraction
	years		pounds	months		per cent	per cent	gram per 100 cc.	reading
Brown.....	8	Grade Jersey	3	9	1.0354	14.88	5.00	0.898	45.2
Nigger.....	8	Grade Jersey	8	6	1.0344	15.11	5.40	0.876	43.6
Grandma.....	10	Holstein	8	$\frac{1}{2}$	1.0317	15.16	6.00	0.805	44.2
Hazel.....	3	Grade Jersey	4 $\frac{1}{2}$	2	1.0328	14.95	5.60	0.788	43.4
Spot.....	5	Grade Jersey	7	5	1.0315	15.71	6.50	0.744	43.1
Rabbit.....	7	Grade Jersey	8	2	1.0325	14.63	5.40	0.787	43.9
Silver.....	3	Grade Jersey	4 $\frac{1}{2}$	2	1.0302	14.89	6.10	0.707	42.3
Baby.....	6	Jersey	7	2	1.0260	8.20	0.880	43.1
Goldie.....	12	Jersey	5	6	1.0298	15.64	6.80	0.840	43.4
Red.....	8	Grade Jersey	5	6	1.0295	13.15	4.80	0.842	41.5
Brownie.....	5	Brown Grade Jersey	9	5	1.0298	15.40	6.60	0.844	44.4
Rose.....	10	Red Grade Jersey	8 $\frac{1}{2}$	8	1.0293	13.82	5.40	0.874	43.6
Jessie.....	6	Jersey	12 $\frac{1}{2}$	2	1.0315	14.14	5.20	0.744	42.0
Mollie.....	9	Holstein	6	10	1.0316	11.75	3.20	0.813	42.0
Marie.....	7	Jersey	5	10	1.0321	7.40	0.902	43.6
White.....	8	Grade Jersey	8	5	1.0351	14.57	4.80	0.866	43.6
Cherry.....	8	Grade Durham	9	4	1.0329	14.01	4.80	0.795	44.2
Crip.....	5	Grade Jersey	6	2	1.0322	14.55	5.40	0.820	41.7
Juliet.....	8	Grade Jersey	5 $\frac{1}{2}$	4	1.0345	11.52	2.40	0.708	42.8
Composite ²	29	1.0302	15.01	6.20	0.810	43.4
Herd ³	129 $\frac{1}{2}$	1.0315	13.89	5.00	0.805	42.8

¹ U. S. Bur. Animal Industry Bull. 134, p. 23.² Composite of 6 Grade Jerseys.³ Four Jerseys; 12 Grade Jerseys; 2 Holsteins; 1 Durham.

TABLE 7.

*Composition of 8 samples of herd milk.
(Arranged in order of Total Solids.)*

TOTAL SOLIDS	FAT	SOLIDS NOT FAT	PRO-TEIN	SUGAR	COPPER SERUM ^M		SOUR SERUM		
					Ash	Refrac- tion	Ash	Refrac- tion	
<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>gram per 100 cc.</i>	<i>reading</i>	<i>gram per 100 cc.</i>	<i>reading</i>	
13.02	4.60	8.42	0.790	40.7	
12.95	4.10	8.85	3.29	4.74	0.70	37.4	0.774	40.4	
12.62	3.40	9.22	3.04	5.01	0.76	38.5	0.816	41.0	
12.60	3.35	9.25	2.33	5.04	0.78	38.7	0.832	41.9	
12.57	4.00	8.57	3.18	4.82	0.68	37.2	0.766	41.5	
12.24	3.55	8.69	3.34	4.65	0.74	36.7	0.808	
12.13	3.35	8.78	2.96	4.72	0.80	36.8	41.3	
11.67	3.40	8.27	2.84	4.51	0.77	36.3	0.804	39.1	
Maximum:	13.02	4.60	9.25	3.34	5.04	0.80	38.7	0.832	41.9
Minimum:	11.67	3.35	8.27	2.33	4.51	0.68	36.3	0.766	39.1
Average:	12.48	3.72	8.76	3.00	4.78	0.75	37.4	0.799	40.8
<i>Average of 67 samples from herds reported in 1914.</i>									
12.83	4.06	8.77	37.9	0.798	41.7	

There is no relation between the refraction of the sour serum and the sour serum ash, since these figures depend upon different milk constituents. When both of these figures fall below the lowest limits established for pure milk (38.3 and 0.730) it is absolute proof of the presence of added water, and all possibility of the sample being abnormal milk from a sick cow is removed.

In the determination of the ash of sour serum there are less than 2 grams of organic matter to be burned, and the influence of combustion upon the 190 mg. of ash is very slight.

The refractive index of the sour serum occurs half way between the copper and acetic indices. Where a milk has partially soured the copper serum reading will be higher, and the acetic serum lower than would be given by the same milk before souring. In such cases the sour serum is the only reliable reading to be made.

Six years' experience with the refractive index and 4 with the sour serum ash have shown these methods to be invaluable. In all doubtful cases the sour serum ash has served as a court of last resort.

RECOMMENDATIONS.

It is recommended—

- (1) That the following be adopted as auxiliary provisional methods.

DETECTION OF ADDED WATER.

*Sour serum*¹.

Allow the milk to sour spontaneously, filter and determine the index of refraction of the clear serum by means of the Zeiss immersion refractometer. A reading below 38.3 indicates added water.

*Ash of sour serum*².

Allow the milk to sour spontaneously and filter. Transfer 25 cc. of this serum to a flat-bottomed platinum dish and evaporate to dryness over the water bath. Heat the contents of the dish over a small flame (to avoid sputtering) until charred. Place the dish in an electric muffle, with pyrometer connected, and ash at a heat not greater than 500°C., or 900°F. Cool and weigh. Express result in grams per 100 cc. An ash below 0.730 indicates added water. (A white ash is invariably obtained after one leaching.)

Ash of acetic serum.

Transfer 25 cc. of the serum to a flat-bottomed platinum dish and proceed as directed under "Ash of Sour Serum". An ash figure below 0.715 gram per 100 cc. indicates added water.

(2) That in conjunction with the copper, acetic or sour serum refraction method, the ash of the sour serum or of the acetic serum be determined in all cases where the indices of refraction fall below the minimum limit. The acetic serum ash multiplied by the factor 1.021 equals the sour serum ash (dilution of the acetic serum being 2%).

No report was made by the referee on the general subject of food adulteration.

REPORT ON COLORS.

By W. E. MATHEWSON (Food and Drug Inspection Laboratory, U. S. Appraiser's Stores, New York, N. Y.), *Associate Referee*.

Two samples of commercial caramel and sealed samples of high grade commercial amaranth, orange I, naphthol yellow S, and light green S F yellowish were sent out for collaborative work. The collaborating analysts were asked to examine the caramels in 0.2% solution by those methods for the detection of this substance that they consider best. In the coal tar dyes they were asked to determine the amount of color by the titanium trichlorid reduction method of Knecht and Hibbert which has the merit of using a comparatively simple reagent, stable if protected from the air, and which has given good results in the New York Food and Drug Inspection Laboratory of the U. S. Bureau of Chemistry when used under

¹ Matthes and Müller. Z. öffent. Chem., 1903, 9: 173.

² Burr and Berberich. Chem. Ztg., 1908, 32: 617-8.

the proper precautions and with a thorough consciousness of the sensitiveness of the reagent to air. With the samples was sent a statement of the precautions to be followed in applying the method to each of the permitted dyes. These are embodied in the suggested provisional methods which come before the Association this year and need not be given here.

The collaborators reported upon the caramel samples as follows:

RESULTS OF COLLABORATIVE WORK.

C. F. Jablon: The samples of caramel under observation did not behave alike to the Amthor test. In solution of 0.2%, one gave a slight precipitate, while the other gave a pronounced precipitate. In stronger solutions (1%) a slight turbidity was observed in the first, while the other showed a pronounced precipitate.

The Woodman-Newhall test does not appear to give any more reliable results. In weaker solutions (0.2%) it appears to be less satisfactory, while a 1% solution gave positive reactions. To sum up the series of experiments, it is safe to state that although strong caramel solutions gave positive tests, ordinary concentrations, such as may be found in food analysis, do not give very satisfactory results.

Ray W. Clough: A few qualitative tests on the caramel samples gave the following results:

Crampton and Simons fuller's earth method¹, all color extracted from a 0.2% solution; Amthor test, negative; Woodman-Newhall method, good results (especially heavy precipitate with the phenylhydrazin reagent).

C. R. Smith: The Woodman-Newhall method was applied to the samples of caramel. It has been found that they, as well as other samples of commercial caramel and caramel prepared in the laboratory, precipitate out well in the Woodman-Newhall method. It is doubtful if the same holds good in fruit juices and other food products. In testing black currant juice of high acidity no precipitate was produced, using the usual proportion of reagents after neutralizing with potash.

By adding a large excess of zinc chlorid (8 cc.) and potash sufficient to produce a good precipitate a large amount of brownish black coloring matter was carried down, which, however, did not precipitate by the paraldehyde test. The Woodman-Newhall test applied with special reference to each substance may be useful if carefully studied and developed.

The results obtained by the collaborators in the determination of the amount of color in the samples of commercial anilin colors are given in Table 1.

¹ U. S. Bur. Chem. Bull. 107, Rev., p. 199.

TABLE 1.

Determinations of the amount of pure color in samples of anilin colors by titration with titanium trichlorid.

ANALYST	NAPHTHOL YELLOW S	ORANGE I	AMARANTH	LIGHT GREEN S F YELLOWISH
	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>
C. L. Black, Food and Drug Inspection Laboratory, U. S. Appraiser's Stores, Philadelphia, Pa.....	93.28	90.82	88.39	66.44
A. L. Burns, Food and Drug Inspection Laboratory, U. S. Appraiser's Stores, New York, N. Y.....	92.9	91.7	88.6	66.8
C. F. Jablon, Food and Drug Inspection Laboratory, U. S. Appraiser's Stores, New York, N. Y.....	92.7	91.0	88.7	67.1
Ray W. Clough, Arcade Annex Building, Seattle, Wash.....	93.4	93.8	88.8	66.1
C. R. Smith, Food and Drug Inspection Laboratory, U. S. Appraiser's Stores, New York, N. Y.....	93.1	93.2	89.1	67.9

THE SEPARATION OF PONCEAU 3R FROM OTHER DYES, ESPECIALLY
NAPHTHOL YELLOW S BY THE USE OF A
SOLUBLE BARIUM SALT.

(C. F. Jablon, New York.)

As the quantitative separation and estimation of ponceau 3R and naphthol yellow S is, under ordinary conditions, rather slow, a method was sought whereby the more or less tedious amyl alcohol extraction would be eliminated.

Inasmuch as ponceau 3R forms a very difficultly soluble barium compound¹ a series of experiments were performed using barium and strontium salts as precipitants in solutions containing the dyes in various concentrations. The best results were obtained as follows:

Dissolve the dye mixture in a definite amount of water. Transfer an aliquot containing about 0.1–0.3 gram of naphthol yellow S to a 250 cc. volumetric flask and dilute with water to about 225 cc. Add 2.0 cc. of 10% barium acetate and 1 cc. of glacial acetic acid, shake thoroughly and make up to the mark with water. Then pass through a dry filter and titrate an aliquot with standard titanium trichlorid solution in sodium tartrate solution, using light green S F yellowish as indicator. Titrating an equivalent part of the original solution gives the total color, and subtracting the yellow, the difference gives the value for ponceau 3R.

Table 2 gives some results obtained.

¹ Heumann. Die Anilinfarben und ihre Fabrikation. 1903, 4 (I): 856.

TABLE 2.

Determinations by the barium method of naphthol yellow S in presence of ponceau 3R.
(C. F. Jablon, New York.)

SAMPLE	WEIGHT PONCEAU TAKEN	WEIGHT YELLOW TAKEN	WEIGHT, YELLOW FOUND	YELLOW RECOVERED
	gram	gram	gram	per cent
1	0.100	0.2491	0.2496	100.2
2	0.200	0.2076	0.2050	98.7
3	0.300	0.2030	0.2028	99.9
4	0.200	0.1833	0.1819	99.2
5	0.100	0.1538	0.1555	101.2
6	0.300	0.1005	0.0998	99.0
7	0.200	0.0240	0.0258	107.6
8	0.300	0.0000	0.0013

The filtrate from the precipitated ponceau 3R was somewhat redder in shade, so it was deemed advisable to determine colorimetrically with a Koenig-Martens instrument the amount of ponceau remaining in solution. Following are the amounts found, using various concentrations of ponceau 3R:

Weight of ponceau taken	Ponceau in solution
gram	per cent
0.05	2.25
0.125	1.4
0.250	0.5
0.500	0.2
1.000	0.1

The incomplete precipitation is probably due to the fact that the ponceau 3R used was not a chemically uniform product. It was the ordinary grade of dye used for food products which contains more or less of the lower homologues derived from xyloidin, etc., and also small amounts of monosulphonated color such as sodium trimethyl-benzene-azo-2-naphthol-6-sulphonate.

Mixtures of ponceau 3R with the other permitted dyes in amounts varying from 0.1–0.3 gram of each dye when treated by the procedure just described showed that erythrosine is precipitated almost completely, and the indigo somewhat less so with ponceau. Amaranth and orange I are carried down with the ponceau precipitate only slightly, if at all. Light green S F yellowish appears to remain entirely in the filtrate.

Because of the high relative oxidizing power of naphthol yellow S as shown towards titanium trichlorid, the error caused by the presence of a small and varying amount of ponceau in the filtrate does not greatly affect the determination of the yellow with mixtures containing not too small amounts of the latter. With the other dyes this is not the case, but the results stated may be applied to the analysis of mixtures containing ponceau and naphthol yellow with the others. Here the total dye

in the filtrate is determined with titanium, the amaranth, green, etc., estimated directly by some suitable method, and correction made to obtain the value for the yellow. With lesser accuracy, the procedure may be used for the direct separation of ponceau from orange, amaranth and green. The quantitative determination of indigo carmine in admixture with amaranth by reduction with titanium trichlorid, reoxidation with air, and estimation of the blue colorimetrically seems to give good results.

Ponceau and yellow being separated with some difficulty by other methods and often used in admixture with each other, the procedure just given will be found useful in examining mixtures.

THE USE OF THE SPECTROPHOTOMETER FOR THE EXAMINATION OF FOOD COLORING MATTER.

(W. E. Mathewson, New York.)

No other methods have the wide range of applicability of those depending on exact measurements under suitable conditions of the opacity of solutions to light of various wave lengths. The principles and methods of spectrophotometry have been developed by numerous investigators and are discussed in an extensive literature¹. These methods are in limited use, probably because the necessary instruments are so expensive that few laboratories are equipped with them. Hence no collaborative work could be done this year. In this part of the report some experimental work of the referee is reported which was designed to learn to what extent spectrophotometric methods might be used in the analysis of food colors. As each of the various optical systems that have been devised and each of the light sources available has its advantages and disadvantages, such apparatus as seemed most suitable was selected.

APPARATUS AND PROCEDURE.

The spectrophotometer used was a Koenig-Martens instrument with Rutherford prism, made by Schmidt and Haensch. Some experience with a spectrophotometer of the Vierort principle has convinced the writer that those forms of apparatus in which an eye-piece is employed (forming a system focused on the collimator slit) are much more difficult to use in work where it is very frequently necessary to take readings in parts of the spectrum in which the absorption varies greatly for small variations in the wave length. As a light source a 1500-3000 c. p. projection lamp with automatic arc and condensing lenses was employed. A Nernst

¹ For a résumé compare especially Kayser. *Handbuch der Spectroscopie*. 1905, 3; and G. and H. Kruss. *Kolorimetrie und Quantitative Spektrolana yse*. 2d ed., 1909.

lamp although steadier can scarcely be used for measurements in the blue and violet. Monochromatic light sources suitable for all regions of the spectrum would be much more preferable, but would add greatly to the cost of the equipment. An absorption cell of the Vierort type was used, 11 mm. in width and containing the Schultz glass cube exactly 1 cm. in thickness. This form of cell is rather objectionable for measurements in non-homogeneous light, as all the rays passing into the instrument must go through 1 mm. of the solution; but the laboratory is not provided with the better apparatus devised by Martens and Grünbaum¹. The cell was placed before the instrument, the glass cube first in front of one slit, then the other. The ocular slit or screen in the telescope is rather troublesome to get at and adjust in the Koenig-Martens instrument, and chiefly for this reason was set at a width corresponding to 100 (smallest) divisions on the scale carrying the telescope (at the mercury line 546.1), and kept at this width for all the measurements. To correspond, the telescope was moved 100 scale divisions between the successive regions. The jaws of the divided slit before the collimating lens were set as close as possible, still giving a field of sufficient brightness, and the observations made with the glass cube before each half of the slit after each resetting of the latter. The necessary opening of the collimator slit corresponded to a maximum overlapping in the field of view of rays differing by about $2.5 \mu\mu$ in the indigo and violet to $0.1 \mu\mu$ in the greenish yellow. In all cases the width of the slit was noted in taking measurements, though for Table 3, containing averaged figures, the statements just made are sufficient. For each reading the rotating nicol prism was set 2 or 3 times and the mean value recorded. The results were calculated to "extinction coefficients" e , by the formula

$$e = \log \tan a_1 + \log \cot a_2$$

a_1 and a_2 representing respectively the angular readings with the Schulz body before the right and left parts of the slit. For the loss of light by reflection at the surfaces of the Schulz body the correction for water was found to be 0.02, this amount to be added to the observed "extinction coefficient". In Table 3, this correction has not been made, however.

EXPERIMENTAL RESULTS.

Extinction coefficients of the permitted dyes.—In Table 3 are given the observed "specific extinction coefficients" obtained with solutions of the 7 permitted dyes, corresponding to the values for 0.001% solutions. Solutions of each color were made containing 0.001%, 0.004%, 0.008%, etc., of pure dye as determined by analysis, the strongest solution being

¹ Ann. Physik., 1903, 4th ser., 12: 991-2.

from 0.064–0.512%. The extinction coefficients for each solution were determined in those regions where the values ranged from 0.030–1.50. From these figures the e values for 0.001% solutions through the whole spectrum were calculated, it being assumed that the observed figures of value equal to about 0.60, if not more accurate, were at least more suitable for subsequent application.

Light green S F yellowish being quite sensitive to alkali, dilute solutions in pure water if kept in glass soon became paler and in 24 hours may show a conversion of 50% or more of the dye into the colorless carbinol form. Orange I and erythrosine are also sensitive indicators and may contain as impurities small amounts of sodium carbonate, free color acid, etc. This source of difficulty was practically eliminated by adding to each solution a little of a mixture of acetic acid and secondary sodium phosphate. A stock solution was made containing in each liter 71.4 grams of crystallized secondary sodium phosphate and 9.6 cc. of glacial acetic acid. Five cc. of this solution were used in making up each 100 cc. of the dye solutions, giving these latter a disodium phosphate normality of 0.01, and an acetic acid normality of 0.008 plus. This amphoteric mixture is faintly acid to litmus, faintly alkaline to alizarin and does not perceptibly change the absorption of the solutions of the colors other than light green S F yellowish, when added to their absolutely neutral solutions in not too large quantities.

The extinction coefficients as determined are not exactly proportional to concentration, being affected by a number of factors. The molecular condition of both electrolytes and colloids varies with changes in concentration, and the use of non-homogeneous light is open to various objections. However, the figures serve as a guide, indicating especially where are located those regions of maximum absorption best adapted for measurements, and the values to be expected. The accurate estimation of a dye must be made by determining the approximate concentration of its solution by the use of Table 3, changing the concentration, if necessary, so that the values for e will be from 0.6–1.00 in the region of maximum absorption, making a standard solution of the same calculated concentration from a pure color and determining the absorption constants for each under exactly the same conditions. The values obtained for e will not differ greatly and it may be assumed under these conditions that the differences are proportional to concentration.

Analysis of mixtures.—The analysis of mixtures must be carried out in a similar way. After quantitative examination of the constituents by chemical, spectroscopic or other means, measurements are made in those regions where the constituent dyes show the maximum absorption. The concentration is calculated, using the figures in Table 3, and from this a similar standard solution is prepared with colors of known purity. This is

TABLE 3.

Specific extinction coefficients of the 7 permitted food dyes.

(0.01 gram per liter is taken as unit concentration; i.e., the numbers given represent the coefficients of 0.001% solutions.)

WAVE LENGTH OF LIGHT IN REGION MEASURED	SCALE READ- INGS	NAPHTHOL YELLOW S	ORANGE I	PONCEAU 3R (FROM COMMER- CIAL PSEUDO- CUMIDIN)	ERY- THROSINE	AMA- RANTH	INDIGO- CARMINE	LIGHT GREEN SF YELLOW- ISH
418.8-422.6	22	0.46	0.30	0.13	0.027	0.093	0.029	0.18
422.6-426.5	23	0.46	0.32	0.13	0.028	0.095	0.029	0.18
426.5-430.7	24	0.46	0.38	0.13	0.028	0.105	0.030	0.18
430.7-435.1	25	0.45	0.45	0.13	0.029	0.11	0.031	0.17
435.1-440.0	26	0.45	0.52	0.14	0.030	0.115	0.032	0.15
440.0-444.5	27	0.44	0.58	0.155	0.034	0.125	0.033	0.13
444.5-449.6	28	0.40	0.67	0.17	0.040	0.13	0.034	0.105
449.6-455.0	29	0.34	0.73	0.19	0.052	0.14	0.034	0.077
455.0-460.7	30	0.29	0.80	0.23	0.068	0.165	0.034	0.056
460.7-466.7	31	0.23	0.87	0.265	0.085	0.19	0.034	0.034
466.7-473.1	32	0.16	0.91	0.30	0.12	0.215	0.034	0.023
473.1-480.0	33	0.105	0.93	0.34	0.17	0.25	0.034	0.015
480.0-487.1	34	0.060	0.90	0.39	0.24	0.29	0.035	0.011
487.1-494.8	35	0.031	0.84	0.43	0.32	0.33	0.038	0.011
494.8-503.0	36	0.014	0.71	0.48	0.37	0.37	0.043	0.013
503.0-511.8	37	0.0051	0.59	0.48	0.48	0.40	0.053	0.017
511.8-521.2	38	0.0024	0.43	0.46	0.76	0.41	0.066	0.024
521.2-531.4	39	0.00092	0.27	0.425	0.97	0.41	0.090	0.036
531.4-542.3	40	0.00031	0.17	0.37	0.59	0.38	0.125	0.055
542.3-554.2	41	0.00005	0.092	0.24	0.16	0.32	0.17	0.066
554.2-567.1	42	Less	0.043	0.11	0.032	0.24	0.23	0.14
567.1-581.1	43	0.019	0.045	0.010	0.14	0.30	0.23
581.1-596.4	44	0.0072	0.017	0.0023	0.054	0.37	0.36
596.4-613.3	45	0.0024	0.0056	0.0004	0.016	0.46	0.53
613.3-631.9	46	0.0008	0.0018	0.0001	0.0046	0.41	0.81
631.9-652.5	47	0.0003	0.0004	0.0013	0.023	0.77
652.5-675.6	48	0.0001	0.0001	0.0003	0.075	0.30
675.6-701.4	49	0.0002	0.022	0.069
701.4-730.6	50	0.007	0.010
622.4-642.0	46.5	0.88

examined under the same conditions. If, as should be the case, the ϵ values do not differ more than 10%, the figures given in Table 3 are shown to be sufficiently correct under the applied conditions to be used for calculating the corrections to be made, these being deduced from the figures in the table and the differences between the ϵ values for the known and the unknown solutions. With mixtures, especially, the use of non-homogeneous light is a cause of differences and useful results can hardly be expected without comparative solutions. The figures given for light in the violet, of wave length less than $430\mu\mu$, are quite inaccurate, and for analytical purposes it would be scarcely advisable to make measurements beyond $440\mu\mu$.

Most binary mixtures may be analyzed with fair ease and accuracy. With more complicated ones, it has been found that usually one or more

of the constituents may be determined with advantage in this way, but that the analysis is best made partly by other methods.

In Table 4 are given the results of 5 consecutive test analyses of mixtures of 2 components.

TABLE 4.
Spectrophotometric analysis of mixtures of 2 dyes.
(W. E. Mathewson, New York.)

DYES	CONCENTRATION GRAM PER 100 CC.	PER CENT OF TRUE VALUE FOUND BY FIRST CALCU- LATION (USING FIGURES OF TA- BLE 3)	PER CENT OF TRUE VALUE FOUND AFTER MAKING COMPARISON SO- LUTIONS AND DE- TERMINING COR- RECTION
Naphthol yellow S.....	0.005625	101.0	101.7
Light green S F yellowish.....	0.002375	95.6	100.4
Indigo carmine.....	0.001800	92.6	99.1
Amaranth.....	0.002200	101.0	100.7
Orange I.....	0.002000	106.0	102.0
Ponceau 3R.....	0.001440	94.4	96.7
Orange I.....	0.000800	99.0	101.0
Naphthol yellow S.....	0.007200	100.1	99.0
Amaranth.....	0.002000	97.8	98.4
Erythrosine.....	0.001200	102.3	100.0

Maximum error 3.3%; average error 1.3%.

The calculations with binary mixtures may be made more easily graphically than algebraically.

Determination of coal tar dyes other than those permitted.—The optical method permits a good quantitative estimation with extremely small amounts of color, and has been found to be well suited for the determination of such non-permitted dyes as orange II, acid yellow G, tartrazine and rose bengal in food products.

Determination of natural coloring matters used for food products.—Very few of these coloring matters can be estimated even approximately by chemical methods. Even where the coloring principle has never been isolated in purity, the method still allows an expression of the amount of color in terms of the extinction coefficients and permits standards to be set for such food coloring products as caramel, archil, etc.

A number of samples of saffron examined in the New York Food and Drug Inspection Laboratory of the U. S. Bureau of Chemistry have shown a wide variation in tinctorial power. One gram of the drug was macerated with occasional shaking for 24 hours with 200 cc. of alcohol, exactly 50% by volume. Two cc. of this solution were measured off, diluted to 100 cc. with 50% alcohol, and the values for e determined. The value of the extinction coefficient at 461–467 μ varied from 0.72–1.73.

A few analyses have indicated that curcumine in turmeric root is readily determined by extraction of the powdered drug with 95% alcohol in a Soxhlet apparatus, dilution of the extract so that the ratio to the weight of the turmeric taken is about 10,000: 2 and determination of the extinction coefficients of the solution. These may be compared with the values obtained with pure crystallized curcumine under the same conditions.

REPORT ON SACCHARINE PRODUCTS.

DETECTION OF ARTIFICIAL INVERT SUGAR IN HONEY.

By F. L. SHANNON (Dairy and Food Department, Lansing, Mich.),
Associate Referee.

The work this year was a continuation of work previously inaugurated. Last year the referee selected 9 of the tests the literature showed to be most commonly used. These 9 tests were submitted to the collaborators along with samples and each test studied. From this work the referee for 1914 recommended "That four of the tests which proved the most satisfactory in the hands of all the collaborators be further studied". Accordingly instructions for these 4 tests were sent with the samples to the collaborators.

PREPARATION OF SAMPLES.

Seven samples were prepared as follows:

Sample A.—Pure honey plus 3% commercial invert sugar¹.

Sample B.—Pure honey plus 5% commercial invert sugar.

Sample C.—Pure honey plus 10% commercial invert sugar.

Sample D.—Pure honey.

Sample E.—Pure honey plus 3% Neumoline².

Sample F.—Pure honey plus 5% Neumoline.

Sample G.—Pure honey plus 10% Neumoline.

INSTRUCTIONS TO COLLABORATORS.

It is requested that the following tests be applied to the samples submitted:

FIEHE'S ORIGINAL TEST³.

Reagent.—Dissolve 1 gram of resorcinol in 100 cc. of HCl (1.19). Redistilled ether.

Manipulation.—Rub 1 gram of honey in a mortar with ether. Filter off the ether and evaporate at room temperature. Moisten the residue with a drop of the reagent.

Results.—In the presence of artificial invert sugar an orange-red color is developed, changing to cherry-red and then to brown-red. Pure honeys sometimes give a pink coloration. Note color after standing 10 minutes and again after standing 24 hours.

¹ Obtained on the market and sold as such.

² A commercial product put on the market as a non-crystallizable sirup and also a substitute for honey.

³ Analyst, 1908, 33: 397.

HARTMANN'S MODIFICATION OF FIEHE'S TEST¹.

Reagent.—Same as original Fiehe Test.

Manipulation.—Add 2 drops of the reagent directly to 1 gram of the honey in a porcelain dish.

Results.—If artificial invert sugar is present, a cherry-red color appears as in the original test. Natural honeys give the reaction after standing about 45 minutes.

BRYAN'S MODIFICATION OF FIEHE'S TEST².

Reagent.—Same as original Fiehe Test.

Manipulation.—Place 10 cc. of a 50% honey solution in a test tube and run in 5 cc. of ether on top. Shake contents gently and allow to stand for some time until the ether layer is perfectly clear; transfer 2 cc. of this clear ether solution to a small test tube and add a large sized drop of the resorcin solution. Shake and note immediately the color.

Results.—In the presence of artificial invert sugar the drop of added acid in the bottom assumes immediately an orange-red color, turning to a dark red.

FEDER'S ANILIN CHLORID TEST³.

Reagent (freshly prepared).—To 100 cc. of C. P. anilin add 30 cc. of 25% hydrochloric acid.

Manipulation.—Mix directly 5 grams of the honey in a porcelain dish with 2.5 cc. of the anilin reagent.

Results.—Bright red color indicates the presence of artificial invert sugar. The intensity of the color is proportional to the amount present.

Give your opinion, based on the foregoing tests, as to which samples are adulterated, if any, and which are pure, if any. Report results on each test on each sample as positive, negative or doubtful.

TABLE 1.
Results of collaborators on Sample A.
(Pure honey plus 3% commercial invert sugar.)

ANALYST	FIEHE'S ORIGINAL TEST		HARTMANN'S MODIFICATION OF FIEHE'S TEST		BRYAN'S MODIFICATION OF FIEHE'S TEST	FEDER'S ANILIN CHLORID TEST
	10 minutes	24 hours	10 minutes	24 hours		
S. F. Sherwood, Bureau of Chemistry, Washington, D. C.....	Very strong trace	Very strong trace	Trace	Very dark cherry-red	Trace	Very slight trace
J. O. Clark, Department of Agriculture, Atlanta, Ga.....	Positive	Positive	Positive	Positive	Positive	Positive
W. L. Scovill, Dairy and Food Department, Lansing, Mich.....	Positive	Positive	Positive	Positive	Positive	Positive
N. A. Childs, Dairy and Food Department, Lansing, Mich.....	Positive	Positive	Positive	Positive	Positive	Positive
F. L. Shannon, Dairy and Food Department, Lansing, Mich.	Positive	Positive	Positive	Positive	Positive	Positive
C. G. Sutton, Dairy and Food Department, St. Paul, Minn.....	Positive	Positive	Positive	Positive	Positive	Positive

¹ Z. Nahr. Genussm., 1911, 21: 374.

² U. S. Bur. Chem. Bull. 154, p. 15.

³ Analyst, 1911, 36: 586.

TABLE 2.

Results of collaborators on Sample B.
(Pure honey plus 5% commercial invert sugar.)

ANALYST	FIEBE'S ORIGINAL TEST		HARTMANN'S MODIFICATION OF FIEBE'S TEST		BRYAN'S MODIFICATION OF FIEBE'S TEST	FEDER'S ANILIN CHLORID TEST
	10 minutes	24 hours	10 minutes	24 hours		
S. F. Sherwood.....	Very strong trace	Very strong trace	Strong trace	Very dark cherry-red	Strong trace	Trace
J. O. Clarke.....	Positive	Positive	Positive	Positive	Positive	Positive
W. L. Scovill.....	Positive	Positive	Positive	Positive	Positive	Positive
N. A. Childs.....	Positive	Positive	Positive	Positive	Positive	Positive
F. L. Shannon.....	Positive	Positive	Positive	Positive	Positive	Positive
C. G. Sutton.....	Positive	Positive	Positive	Positive	Positive	Positive

TABLE 3.

Results of collaborators on Sample C.
(Pure honey plus 10% commercial invert sugar.)

ANALYST	FIEBE'S ORIGINAL TEST		HARTMANN'S MODIFICATION OF FIEBE'S TEST		BRYAN'S MODIFICATION OF FIEBE'S TEST	FEDER'S ANILIN CHLORID TEST
	10 minutes	24 hours	10 minutes	24 hours		
S. F. Sherwood.....	Very strong trace	Very strong trace	Very strong trace	Very dark red	Very strong trace	Very strong trace
J. O. Clarke.....	Positive	Positive	Positive	Positive	Positive	Positive
W. L. Scovill.....	Positive	Positive	Positive	Positive	Positive	Positive
N. A. Childs.....	Positive	Positive	Positive	Positive	Positive	Positive
F. L. Shannon.....	Positive	Positive	Positive	Positive	Positive	Positive
C. G. Sutton.....	Positive	Positive	Positive	Positive	Positive	Positive

TABLE 4.

Results of collaborators on Sample D.
(Pure honey.)

ANALYST	FIEBE'S ORIGINAL TEST		HARTMANN'S MODIFICATION OF FIEBE'S TEST		BRYAN'S MODIFICATION OF FIEBE'S TEST	FEDER'S ANILIN CHLORID TEST
	10 minutes	24 hours	10 minutes	24 hours		
S. F. Sherwood....	Negative	Doubtful	Negative	Very dark red	Negative	Negative
J. O. Clarke.....	Negative	Negative	Slight pink Doubtful	Positive	Negative	Negative
W. L. Scovill.....	Negative	Positive	Negative	Positive	Negative	Negative
N. A. Childs.....	Negative	Negative	Doubtful	Positive	Negative	Negative
F. L. Shannon.....	Negative	Negative	Negative	Positive	Negative	Negative
C. G. Sutton.....	Negative	Negative	Positive Faint pink	Positive	Negative	Negative

TABLE 5.
Results of collaborators on Sample E.
(Pure honey plus 3% Neumoline.)

ANALYST	FIEBE'S ORIGINAL TEST		HARTMANN'S MODIFICATION OF FIEBE'S TEST		BRYAN'S MODIFICA- TION OF FIEBE'S TEST	FEDER'S ANILIN CHLORID TEST
	10 minutes	24 hours	10 minutes	24 hours		
S. F. Sherwood....	Negative	Doubtful	Very slight trace	Very dark color	Very slight trace. Several minutes to develop color	Negative
J. O. Clarke.....	Positive	Positive	Positive	Positive	Positive	Positive
W. L. Scovill.....	Doubtful	Positive	Doubtful	Positive	Positive	Positive
N. A. Childs.....	Doubtful	Negative	Doubtful	Positive	Doubtful	Doubtful
F. L. Shannon.....	Negative	Positive	Negative	Positive	Positive	Doubtful
C. G. Sutton.....	Positive	Positive	Positive	Positive	Positive	Positive
	Very faint pink					Light pink

TABLE 6.
Results of collaborators on Sample F.
(Pure honey plus 5% Neumoline.)

ANALYST	FIEBE'S ORIGINAL TEST		HARTMANN'S MODIFICATION OF FIEBE'S TEST		BRYAN'S MODIFICA- TION OF FIEBE'S TEST	FEDER'S ANILIN CHLORID TEST
	10 minutes	24 hours	10 minutes	24 hours		
S. F. Sherwood....	Doubtful	Trace	Very slight trace	Very dark red	Very slight trace	Doubtful
J. O. Clarke.....	Positive	Positive	Positive	Positive	Positive	Positive
W. L. Scovill.....	Positive	Positive	Positive	Positive	Positive	Positive
N. A. Childs.....	Positive	Positive	Positive	Positive	Positive	Positive
F. L. Shannon.....	Positive	Positive	Positive	Positive	Positive	Positive
C. G. Sutton.....	Positive	Positive	Positive	Positive	Positive	Positive
	Faint pink					Pink

TABLE 7.
Results of collaborators on Sample G.
(Pure honey plus 10% Neumoline.)

ANALYST	FIEBE'S ORIGINAL TEST		HARTMANN'S MODIFICATION OF FIEBE'S TEST		BRYAN'S MODIFICA- TION OF FIEBE'S TEST	FEDER'S ANILIN CHLORID TEST
	10 minutes	24 hours	10 minutes	24 hours		
S. F. Sherwood.....	Trace	Strong trace	Trace	Very dark red	Trace	Trace
J. O. Clarke.....	Positive	Positive	Positive	Positive	Positive	Positive
W. L. Scovill.....	Positive	Positive	Positive	Positive	Positive	Positive
N. A. Childs.....	Positive	Positive	Positive	Positive	Positive	Positive
F. L. Shannon.....	Positive	Positive	Positive	Positive	Positive	Positive
C. G. Sutton.....	Positive	Positive	Positive	Positive	Positive	Positive

TABLE 8.
Conclusion as to purity of samples.

ANALYST	SAMPLE A	SAMPLE B	SAMPLE C	SAMPLE D	SAMPLE E	SAMPLE F	SAMPLE G
S. F. Sherwood.....	Adulterated	Adulterated	Adulterated	Pure	Doubtful	Doubtful	Adulterated
J. O. Clarke.....	Adulterated	Adulterated	Adulterated	Pure	Doubtful	Adulterated	Adulterated
W. L. Scovill.....	Adulterated	Adulterated	Adulterated	Pure	Doubtful	Adulterated	Adulterated
N. A. Childs.....	Adulterated	Adulterated	Adulterated	Pure	Doubtful	Adulterated	Adulterated
F. L. Shannon.....	Adulterated	Adulterated	Adulterated	Pure	Pure	Adulterated	Adulterated
C. G. Sutton.....	Adulterated	Adulterated	Adulterated	Pure	Adulterated	Adulterated	Adulterated

COMMENTS OF COLLABORATORS.

S. F. Sherwood: The color developed in Feder's test is very uncertain. Hartmann's modification is liable to give the color reaction in pure honeys and hence be misleading. Bryan's modification is preferable to Fiehe's original test, as a more thorough extraction of the honey is obtained and less time required. The color should be developed at once. Bryan's modification is preferable to any of the others. It has been used by the writer frequently and with satisfactory results.

J. O. Clarke: All reactions on Sample No. 5 were somewhat faint, but sufficient to be classed as "positive".

W. L. Scovill: In Bryan's modification of Fiehe's test it is preferable to add 2 drops of the reagent instead of 1. The 24 hour test with the original Fiehe reaction and Hartmann's modification is not characteristic, as a pure honey will give a positive reaction.

The original Fiehe test and Hartmann's modification give slight positive reactions with pure honey.

F. L. Shannon: There is no advantage in either Fiehe's original or in Hartmann's modification in allowing them to stand 24 hours and then drawing conclusions. The color is well developed in 2 minutes and the best conclusion can be drawn at the end of that time.

DISCUSSION.

The results of the collaborators on these 4 tests indicate that it is not possible in all cases to detect artificial invert sugar in honey when the adulteration is less than 5%. It is also evident that the possibility of detecting artificial invert sugar in honey depends upon the nature of the invert sugar present. None of the collaborators hesitated to pronounce Samples A, B and C adulterated although Sample A contained but 3% adulterant, while in the case of Sample E, which contained but 3% adulterant, all were doubtful as to its purity, although not quite positive that it was adulterated. Sample A contained 3% of one kind of invert sugar and Sample E 3% of another.

All of these reactions depend upon furfural or its derivatives and the intensity of the color developed depends upon the amount of furfural present. This may be large or small, depending upon the manner in which the artificial invert sugar has been made. No accurate conclusions

can be drawn, therefore, from any of the tests as to the amount of the adulterant present.

Nothing is gained by allowing any of the tests to stand for any length of time before drawing conclusions. The color in all instances is developed at once when the adulteration is perceptible.

RECOMMENDATIONS.

It is recommended—

(1) That Bryan's modification of Fiehe's test, as given on page 150, be adopted as provisional.

(2) That Fiehe's test as given on page 149, adopted as provisional, except that the sentence "Note color after standing 10 minutes and again after standing 24 hours", be stricken out.

(3) That Feder's anilin chlorid test, as given on page 150, be adopted as provisional, except that the sentence "The intensity of the color is proportional to the amount present", be stricken out.

The Association adjourned at 11.55 to reassemble at 1 p.m.



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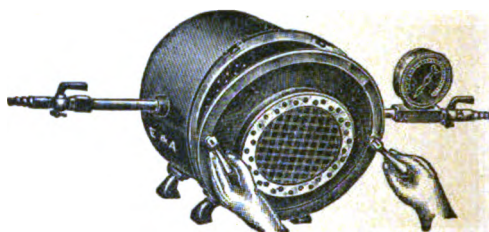
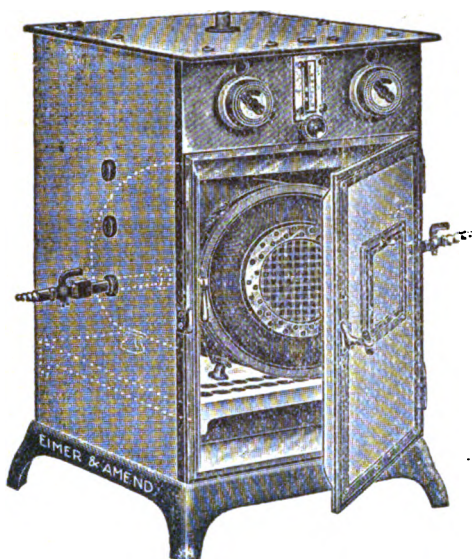
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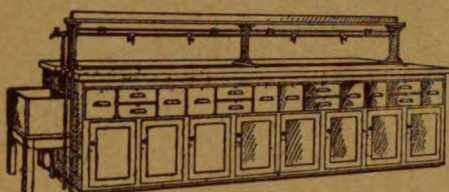
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MONDAY—AFTERNOON SESSION.

REPORT ON FRUIT PRODUCTS.

BY P. B. DUNBAR, *Associate Referee*, AND H. A. LEPPER (Bureau of Chemistry, Washington, D. C.).

The work on fruit products during the past year has been directed principally to a further study of methods for the determination of malic and citric acids. The associate referee for 1914, Mr. H. C. Gore, recommended a continued study of his proposed methods for the determination of these acids, and this recommendation has been followed in the case of the malic-acid method. The present referee has been unable to secure any details of the proposed method for citric acid and consequently no work on this method has been undertaken. A study has been made, however, of the modification of Stahre's method¹ for the determination of citric acid, recently published by Kunz.² The "Methods for the analyses of fruits and fruit products,"³ have also been reviewed, and a critical study of the modified Schmidt-Hiepe method for tartaric, citric, and malic acids⁴ has been made. It has not seemed advisable to send out collaborative samples during the present year, as the details of the methods under study have not been completely worked out.

CITRIC ACID.

A somewhat detailed abstract of the method proposed by Kunz for the determination of citric acid has already been published.⁵ The quantitative method described is applicable only to the determination of citric acid in wines presumably dry. Methods for its qualitative detection in milk, marmalade, and fruit sirup are also described.

The quantitative method as applied to wine consists in treating the concentrated sample with sulphuric acid and bromine water, followed by a solution of potassium bromide. The solution is then oxidized with potassium permanganate, when insoluble pentabromacetone is precipitated quantitatively. While the method appears promising for the determination of citric acid in dry wines and other materials containing small amounts of permanganate reducing substances, it cannot be applied directly

¹ Nordisk Tidskrift (1895), 2: 141; Z. anal. Chem. (1897), 36: 195.

² Arch. Chem. Mikros (1914), 7: 285-299.

³ U. S. Bur. Chem. Bul. 107 (rev.), pp. 77-82.

⁴ Ibid., pp. 80-81.

⁵ Chem. Abstracts (1915), 9: 687.

to fruit juices on account of the presence of large amounts of sugar. It, therefore, becomes necessary in order to apply the method to adopt some procedure for the precipitation of citric acid and its removal from most of the other permanganate reducing bodies. Precipitation as barium citrate from alcoholic solution has been advocated by many investigators, and seems to offer the most practical method of procedure.

The method as finally adopted for study differs little, except for the fact that the citric acid is previously precipitated as barium citrate, from that recommended by Kunz for the determination of citric acid in wine. The procedure used for precipitating the acid is essentially that recommended for malic acid by the previous referee, Mr. Gore, but a solution of barium hydroxid is used instead of the solid substance, and the pectins are not removed before precipitation of barium citrate.

The method is as follows:

Determine the total acidity of the sample. It is convenient to use 1% azolitmin (made by dissolving 1 gram azolitmin in 80 cc. hot water, diluting to 100 cc. with 85% alcohol, and filtering) as an indicator in conjunction with a porcelain spot plate.

Weigh out 25 grams of the sample and transfer to a 600 cc. beaker with 95% alcohol; add an amount of approximately 0.25 N/1 barium hydroxid solution just sufficient to neutralize the acidity of the sample and 5 drops of 50% barium acetate solution, to insure an excess of barium. Dilute to a total volume of 375 cc. with 95% alcohol, stir well and allow to settle. After the precipitates of pectins and barium salts have settled well, decant the supernatant liquid into another beaker. Filter the decanted liquid through a folded filter to remove any small portions of precipitate which it may contain and wash the beaker with 95% alcohol. Filter the remainder of the pectin and barium salt precipitate on the same paper and wash the beaker and paper once with 95% alcohol. Transfer the precipitate quantitatively from the paper to the original beaker with hot water, boil until no more alcohol can be detected by odor, add enough sulphuric acid (1:5) to precipitate all of the barium originally added and to allow 2 cc. of the acid in excess, evaporate by careful boiling to between 60 and 70 cc., cool and add 5 cc. of freshly prepared saturated bromin water; the solution should show an excess of bromin. Transfer quantitatively to a 100 cc. graduated flask and dilute to the mark at room temperature; allow the precipitate of barium sulphate and that caused by the addition of bromin to settle and filter. The precipitate may be separated by centrifuging, and the supernatant liquid decanted if necessary. Now pipette an aliquot of the filtrate, containing not more than 400 mg. of citric acid, calculated from the total acidity of the sample, into a 300 cc. Erlenmeyer flask. The amount of citric acid in the aliquot should, if possible, exceed 50 mg. Add 10 cc. sulphuric acid (1:1) and 5 cc. of potassium bromid solution (15 grams KBr in 40 cc. water). After shaking, warm the flask in a water bath to 48-50°C. and allow it to remain in the bath for five minutes. After removing from the bath, add 25 cc. of 5% KMnO_4 from a burette in rapid drops with frequent interruptions and constant vigorous shaking, care being taken that the temperature during oxidation does not exceed 55°. Set the flask aside until the hydrated peroxid of manganese begins to settle. (The supernatant liquid should be dark brown in color, showing an excess of KMnO_4 . If an excess is not indicated, more KMnO_4 must be added.) Then shake and again set

aside to settle, and repeat this operation until the precipitate takes on a yellow color and most of it has dissolved. Finally, while the solution is still warm, remove the last undissolved portion of the hydrated peroxid of manganese precipitate and also the excess of bromin by adding drop by drop a clear concentrated solution of ferrous sulphate acidified with H_2SO_4 . Allow the solution to cool, with occasional shaking. If the operations have been properly carried out, a heavy white precipitate of pentabromacetone is obtained. This becomes crystalline on occasional shaking and in this condition is entirely insoluble in water. After the precipitate has become crystalline, preferably after standing overnight, collect it by means of gentle suction on a porcelain Gooch crucible provided with a thin pad of asbestos, previously dried over H_2SO_4 in a vacuum desiccator, and wash with distilled water slightly acidified with H_2SO_4 and, finally, twice with distilled water (if the precipitate has a tendency to pass through the filter, 1% H_2SO_4 should be used for washing). Dry the precipitate to constant weight in a vacuum desiccator over H_2SO_4 , protected from strong light. The weight of pentabromacetone multiplied by the factor 0.464 equals citric acid plus 1 molecule of water of crystallization. It sometimes happens that the pentabromacetone is obtained in the form of oily droplets. These also become crystalline on standing, or on cooling, but are usually discolored by negligible traces of manganese or iron. There appears to be no difference in the results obtained whether the precipitate is originally in the form of crystals or oily droplets, which later become crystalline.

The accuracy of this method has been tested by determinations on water solutions of citric acid containing varying amounts of sucrose, and on diluted apple sirup and raspberry sirup containing added known amounts of citric acid, and in some cases added malic and tartaric acids. The results obtained are given in Table 1.

According to Partheil and Hübner,¹ 100 grams of alcohol of specific gravity 0.8092 dissolves 0.00578 gram of barium citrate ($C_6H_4O_7$)₂ Ba₃ + 7H₂O) at 25°C.

In determining small percentages of citric acid, the solubility of barium citrate in the volume of alcohol used will give rise to a relatively large error in the percentage recovered. Considerable time has been given to attempts to obviate this difficulty by determining the citric acid without previous precipitation, but these have been unsuccessful. It is hoped that the difficulty may be met by the use of a solubility correction. It is hardly necessary to point out, however, that a relatively small variation in the actual weight of citric acid found will give a marked variation in the percentage recovered and that comparisons should be made between the actual weights, in grams per 100 cc., of citric acid present and found. From the results on sirups containing added malic and tartaric acids, it appears that these acids do not interfere with the determination. This has been further shown by applying the method to 50 cc. portions of 1% solutions of malic, tartaric, and succinic acids. In no case was a precipitate obtained.

¹ Arch. der Pharm., **241**: 412-435; Chem. Centr. (1903), **74**: (2), 1026.

This method seems of sufficient promise to warrant further study, and it is recommended that the referee for the following year be instructed to take up this work and, if possible, to send out collaborative samples.

TABLE 1.

Determination of citric acid by Kunz modification of Stahre's method.

CITRIC ACID IN AQUEOUS SOLUTION.

NO PRELIMINARY PRECIPITATION AS BARIUM CITRATE.

CITRIC ACID PRESENT		CITRIC ACID RECOVERED		
In 50 cc. sample	Per 100 cc.			
gram	gram	gram	gram per 100 cc.	per cent
0.0184	0.04	0.013	0.03	75.0
0.0908	0.18	0.086	0.17	95.2
0.0923	0.18	0.089	0.18	100.0
0.1845	0.37	0.181	0.36	97.3
0.1853	0.37	0.180	0.36	97.3
0.2270	0.45	0.223	0.45	98.3
0.2779	0.56	0.275	0.55	98.2
0.3706	0.74	0.370	0.74	100.0
0.4632	0.93	0.461	0.92	98.9

PRECIPITATION AS BARIUM CITRATE INCLUDED.

CITRIC ACID PRESENT		APPROXIMATE AMOUNT SUCROSE ADDED PER 100 cc.	CITRIC ACID RECOVERED		
In 25 cc. sample	Per 100 cc.				
gram	grams	grams	grams	grams per 100 cc.	per cent
0.4610	1.84	0	0.4503	1.80	97.68
0.4610	1.84	0.5	0.4491	1.80	97.42
0.4610	1.84	1.0	0.4493	1.80	97.47
0.4610	1.84	2.0	0.4487	1.79	97.33
0.4610	1.84	5.0	0.4465	1.79	96.85
0.4610	1.84	10.0	0.4448	1.78	96.50
0.4610	1.84	20.0	0.4455	1.78	96.64

ADDED CITRIC ACID IN DILUTED APPLE SIRUP.

CITRIC ACID PRESENT		APPROXIMATE MALIC ACID PRESENT	CITRIC ACID RECOVERED		
In 25 cc. sample	Per 100 cc.				
gram	grams	per cent	gram	grams per 100 cc.	per cent
0	0	0.51	0	0	0
0.0369	0.15	0.03	0.029	0.12	80.0
0.0484	0.19	0.53	0.038	0.15	78.9
0.0554	0.22	0.03	0.049	0.20	90.9
0.0727	0.29	0.53	0.059	0.24	82.8
0.0923	0.37	0.03	0.081	0.32	86.5
¹ 0.0969	0.39	0.53	0.082	0.33	84.6
² 0.0969	0.39	0.53	0.082	0.33	84.6
0.1211	0.48	0.53	0.109	0.44	91.7
0.1845	0.74	0.03	0.178	0.71	96.0
0.2707	1.08	0.51	0.243	0.97	89.8
0.4060	1.62	0.51	0.406	1.62	100.0

¹ 2.5 grams sucrose added to 25 cc. sample.

² 5 grams sucrose added to 25 cc. sample.

TABLE 1.—*Continued.*
ADDED CITRIC ACID IN RASPBERRY SIRUP.

CITRIC ACID PRESENT		APPROXIMATE MALIC ACID ADDED	APPROXIMATE TARTARIC ACID ADDED	CITRIC ACID RECOVERED ²		
In 25 cc. sample	Per 100 cc.					
gram	gram	per cent	per cent	gram	gram per 100 cc.	per cent
40.1373	0.55	0	0	0.155	0.62	112.7
40.1373	0.55	0	0	0.154	0.62	112.7
40.0992	0.40	0	0	0.097	0.39	97.5
40.0992	0.40	0.39	0	0.094	0.38	95.0
40.0992	0.40	0	0.40	0.097	0.39	97.5
40.0992	0.40	0.19	0.20	0.093	0.37	92.5

¹ The last four determinations are corrected for the amount of citric acid found in the original sirup by precipitation.

² Calculated from titration of original sirup.

³ Grams of citric acid added in excess of that originally present.

MALIC ACID.

The previous referee, Mr. Gore, has given considerable attention to a study of methods for the determination of malic acid during the past three years. In his report for the year 1912¹ he gives results obtained by eight collaborators, using the polariscopic method proposed by Dunbar and Bacon.² This method depends on the change in the rotation of malic-acid solutions when treated with uranyl acetate. The sample of cider analyzed contained 0.50% of total acidity calculated as malic. The results reported varied from 0.468 to 0.531%, and averaged 0.49%. During the following years, Mr. Gore paid considerable attention to the effects, on the rotations of uranium-malic acid solutions, of various factors. He further investigated the possibility of developing a method based on the change in rotation of malic-acid solutions when treated with ammonium heptomolybdate. As a result of this work he suggested, in his report for 1914, two polariscopic methods for the determination of malic acid, both of which call for a preliminary precipitation of the acid as barium malate.

The uranyl-acetate method suggested differs from the Dunbar-Bacon method, first sent out by Mr. Gore for collaborative work, in that precipitation of malic acid is suggested, a solution of uranyl acetate is used in place of the solid salt, and the amounts of malic acid are determined by referring the polariscopic reading to a table, rather than by the use of a constant factor. The method of Dunbar and Bacon is not applicable in the presence of tartaric acid nor in the case of highly colored liquids which cannot be readily polarized. A method whereby the malic acid may be precipitated and separated from interfering substances is therefore highly desirable. The use of a solution of uranyl acetate in

¹ U. S. Bur. Chem. Bul. 162, p. 63.

² U. S. Bur. Chem. Circ. 76.

place of the solid salt is also advantageous in that it obviates the indefinite error of dilution, due to the solution of unknown amounts of uranyl acetate. In the case of products containing small amounts of malic acid, however, the dilution of the solution with an equal volume of uranyl-acetate solution produces a corresponding decrease in the polarization and thereby increases the relative percentage error due to inaccuracies in reading the polariscope.

The 1914 report on fruit products contained results by only two collaborators using the proposed methods for malic acid suggested by Mr. Gore. These, unfortunately, do not agree very satisfactorily, and it seemed advisable, therefore, to carry out further experimental work with solutions containing known amounts of malic acid. Numerous difficulties were encountered in precipitating the acid; and, although considerable time was given to the work, the results so far obtained are not such as to justify a final recommendation regarding the method. It was found

TABLE 2.
Determination of malic acid by the uranyl-acetate and molybdate methods in aqueous solutions containing tartaric acid.

MALIC ACID PRESENT		TARTARIC ACID PRESENT IN 25 cc. SAMPLE	MALIC ACID RECOVERED					
In 25 cc. sample	Per 100 cc.		By uranyl-acetate method			By molybdate method		
gram	gram	gram	gram	gram per 100 cc.	per cent	gram	gram per 100 cc.	per cent
0.0938	0.38	0.0997	0.092	0.37	97.4	0.102	0.41	107.9
0.0938	0.38	0.1496	0.088	0.35	92.1	0.100	0.40	105.2
0.1407	0.56	0.0997	0.130	0.52	92.9	0.157	0.63	112.5
0.1876	0.75	0.0499	0.165	0.66	98.0	0.223	0.89	118.6

to be difficult, if not impossible, to dissolve the solid barium hydroxid which is added to precipitate the malic acid, and a strong standardized barium-hydroxid solution was therefore substituted. The error due to the solubility of barium malate does not seem to be serious. It was found, however, that in the case of solutions containing malic and tartaric acids, a sufficient amount of barium tartrate was dissolved to reduce the negative polarization somewhat in the uranyl-acetate method and increase it in the molybdate method. The irregularities of results due apparently to this cause are shown in Table 2. Comparison of the actual weights of malic acid present with those found, however, show that the error in grams per 100 cc. is not large. In the case of determinations of malic acid in fruit juices, much of the color of the original sample is carried down with the barium precipitate and again dissolved when this precipitate is taken up in water.

It thus appears that the method suggested for the precipitation of barium malate does not entirely obviate the difficulties in the original method when interfering color is present, but is of promise in determining

malic acid in the presence of tartaric when the color of the solution is not excessive. In the absence of tartaric acid and color the precipitation method introduces a number of time-consuming operations without materially affecting the results.

The work which was undertaken on citric acid prevented an extended study of the malic-acid method. From the results of the collaborative work reported by Mr. Gore in 1912 and those obtained by the present referee and reported in U. S. Bureau of Chemistry Circular 76, it would appear that the malic-acid method as originally described in that circular can undoubtedly be applied with accuracy to solutions which can be polarized readily and which contain no optically active acid other than malic. Where the amount of malic acid present is sufficient, the use of a solution of uranyl acetate is apparently to be preferred to the solid salt. When it is possible to make readings without difficulty, rapid determination may be made with reasonable accuracy without previous neutralization or clarification by polarizing before and after treatment with uranyl acetate. In the presence of tartaric acid, preliminary precipitation as the barium salt is advantageous.

REVIEW OF METHODS FOR THE ANALYSES OF FRUITS AND FRUIT PRODUCTS.

A number of recommendations for changes in the methods for the analyses of fruits and fruit products have been forwarded to the chairman of the committee on the revision of methods. These recommendations will doubtless be included in the report of the committee.

TABLE 3.

Experiments with the Schmidt-Hiepe method for tartaric, citric, and malic acids.¹

NO.	TARTARIC ACID			CITRIC ACID			MALIC ACID		
	Added		Recovered	Added		Recovered	Added		Recovered
	gram	gram		gram	gram	per cent	gram	gram	per cent
1	0.4896	0.4834	98.73	0.4601	0.3955	85.96	0.0304
2	0.4896	0.4884	99.76	0.0019	0.4856	0.4000	82.37
3	0.0195	0.4462	0.2957	66.27	0.4856	0.5832	120.1
4	0.3917	0.3856	98.44	0.3570	0.1760	49.30	0.3885	0.4536	116.8
5	0.1469	0.0706	48.06	0.1339	0.0803	59.97	0.0586
6	0.1469	0.0702	47.78	0.0025	0.1457	0.1286	88.29
7	0.0075	0.1339	0.0141	10.53	0.1457	0.1631	112.0
8	0.0979	0.0957	97.75	0.0920	0.0118	12.83	0.1003	0.0141	13.06
9	0.0490	0.0342	69.80	0.0460	0.0109	23.69	lost
10	0.0490	0	0	0.0102	0.0502	0	0
11	0.0045	0.0460	0.0048	10.43	0.0502	lost
12	0.0490	0.0305	62.24	0.0460	0	0	0.0502	lost

¹ U. S. Bur. Chem. Bul. 107 (rev.), p. 80.

In 3, 7, and 11, where a recovery is recorded for tartaric acid when none was present, the titer was probably caused by calcium acetate retained, due to the limitation to the washing.

The amount of tartaric acid recovered in 3, 7, and 11 is the amount actually titrated, the 0.0286 gram dissolved in the 100 cc. filtrate not being added, as no tartaric was present.

The amount of malic recovered was always corrected for the tartaric (0.0286) present when it was known to be there, due to original addition.

Table 3 contains the results of a study of the modified Schmidt-Hiepe method for the determination of tartaric, citric, and malic acids. From these results it appears that the method is unreliable, and should be dropped as one of the official methods.

RECOMMENDATIONS.

It is recommended—

- (1) That the Kunz modification of Stahre's method for the determination of citric acid be further studied.
- (2) That the uranyl-acetate and ammonium heptomolybdate methods for the determination of malic acid be further studied.
- (3) That the method for "Tartaric, citric, and malic acids (Schmidt-Hiepe method modified) official"¹ be dropped.

REPORT ON WINE.

By B. G. HARTMANN (Bureau of Chemistry Food and Drug Inspection Laboratory, Chicago, Ill.), *Associate Referee*.²

The provisional method for the determination of total tartaric acid content in wines,³ is inaccurate and unreliable. In a paper read before the association in 1912, Hartmann and Eoff called attention to this fact, presenting proof in support of their criticisms and offering for trial a modification of this method. This paper gave the initiative for a series of coöperative investigations extending over a period of three years, conducted for the purpose of determining the relative merits of the two methods as to their accuracy and adaptability to varying conditions. In the course of these investigations the methods were tried on wines, grape juices, and synthetic solutions containing varying amounts of tartaric and phosphoric acids.

The results of this work proved conclusively that the provisional method is far from satisfactory, whereas the proposed method was found to be accurate and reliable.

During the present year five samples of grape juice, with and without the addition of tartaric acid and phosphoric acid, were sent to collaborators with the following instructions:

INSTRUCTIONS TO COLLABORATORS.

The samples were filtered and no precipitation is expected. However, as a precautionary measure, shake the samples thoroughly just before measuring the various portions.

¹ U. S. Bur. Chem. Bul. 107 (rev.), pp. 80-81.

² Presented by M. J. Ingle.

³ U. S. Bur. Chem. Bul. 107 (rev.), p. 86.

ACIDITY OF THE JUICE.

Measure 10 cc. of the sample into a 250 cc. beaker, add 20 cc. distilled water and titrate with tenth-normal sodium hydroxid until two drops of the solution when mixed on a porcelain tile with several drops of a neutral azolitmin solution give no red tint. Prepare the azolitmin solution by dissolving 0.5 gram pure azolitmin in a liter of distilled water, neutralizing with either acid or alkali, as the case may be.

DETERMINATION OF TOTAL TARTARIC ACID.

Provisional method.—Determine as described in U. S. Bureau of Chemistry Bulletin 107 (revised), page 86, using 50 cc. of sample, and dilute with 50 cc. distilled water. Use 20 cc. of alcohol instead of 15 cc. Collect the cream of tartar crystals on a double filter paper in a Büchner funnel (see U. S. Bureau of Chemistry Bulletin 162, p. 73) and titrate with N/10 alkali, using phenolphthalein as indicator. Multiply by 2 to obtain grams per 100 cc. Do not stir longer than 1 minute and do not place in an ice box for the 15 hours prescribed, but allow to remain at room temperature.

Proposed method (Hartmann and Eoff¹).—Transfer 50 cc. of the sample to a 250 cc. beaker and neutralize with sodium hydroxid. The amount of normal alkali required is calculated by multiplying the cc. N/10 sodium hydroxid required to titrate the 10 cc. of the sample (under 1) by 0.5. To the neutralized solution, add enough water to make 100 cc. and add tartaric acid. The tartaric acid used should be of the purest obtainable and should be powdered and dried for about two hours at the temperature of boiling water. Determine the purity of the tartaric acid by titrating 0.30 gram with N/10 alkali, using phenolphthalein as indicator. Keep the tartaric acid in a desiccator. The amount of tartaric acid to be added is calculated by multiplying the number of cc. N/1 NaOH required to neutralize the 50 cc. sample by 0.075. Weigh the amount accurately.

After the tartaric acid has gone into solution, add 15 grams powdered potassium chlorid and stir until the salt has dissolved. Now add 2 cc. glacial acetic acid and 20 cc. of 95% alcohol. Stir until precipitation has started and place in an ice box for at least 15 hours. Collect the cream of tartar crystals on a double filter paper in a Büchner funnel and titrate with N/10 alkali, using phenolphthalein as indicator. Calculate the total tartaric acid and subtract the tartaric added, making proper correction for purity of the latter. This will give the tartaric acid in 50 cc. Multiply by 2 to obtain grams per 100 cc.

The basis of the five samples was a pure Concord grape juice. In order to prevent precipitation of potassium acid tartrate, the filtered juice was diluted with 25% of distilled water. The samples were made up as follows:

No. 1.—The diluted juice.

No. 2.—The diluted juice with the addition of 0.569 gram of tartaric acid per 100 cc.

No. 3.—The diluted juice with the addition of 0.06 gram of phosphoric acid per 100 cc.

No. 4.—The diluted juice with the addition of 0.30 gram of phosphoric acid per 100 cc.

No. 5.—The diluted juice with the addition of 0.086 gram sodium carbonate to neutralize the free tartaric acid.

¹ U. S. Bur. Chem. Bul. 162, p. 72.

All samples were sterilized.

The following results were obtained by the collaborators and the associate referee.

Total tartaric acid (grams per 100 cc.).

ANALYST	SAMPLE									
	1		2		3		4		5	
	No. 1	No. 2	No. 1	No. 2	No. 1	No. 2	No. 1	No. 2	No. 1	No. 2
J. R. Eoff, Jr.	0.528	0.614	0.912	1.210	0.504	0.614	0.414	0.628	0.555	0.624
M. J. Ingle	0.534	0.608	0.924	1.208	0.507	0.620	0.408	0.620	0.558	0.620
F. D. Merrill	0.558	0.627	0.969	1.205	0.542	0.636	0.433	0.638	0.590	0.637
E. H. Berry	0.578	0.628	1.024	1.213	0.579	0.586	0.382	0.586	0.562	0.602
T. G. Gleason	0.556	0.626	0.960	1.200	0.544	0.613	0.444	0.627	0.582	0.615
Edw. F. Higgins	0.564	0.629	0.964	1.205	0.550	0.625	0.450	0.633	0.585	0.615
B. G. Hartmann	0.538	0.618	0.952	1.200	0.526	0.636	0.435	0.644	0.584	0.632
Average	0.554	0.622	0.966	1.203	0.538	0.618	0.427	0.624	0.576	0.619

No. 1, provisional method; No. 2, proposed method.

No comments on the two methods were offered by the collaborators. M. J. Ingle submitted the results which he obtained on the five samples by the Rochelle-salt method, as follows:

The following are the results obtained by using Rochelle salt on the five samples submitted. I did not add any alkali to these determinations, merely a quantity of Rochelle salt calculated by multiplying the number of cc. of N/1 alkali required for complete neutralization by the factor 0.141. The portions of the juice used were measured on July 19 and had to be thoroughly shaken to mix in the precipitated tartar. I then added 50 cc. of water, 2 cc. acetic, 15 grams KCl, and 20 cc. of 95% alcohol, and placed in the ice box over night. I ran these determinations in duplicate and obtained good checks. I found out that one cannot be certain of the state of hydration of these salt crystals, particularly if grinding is resorted to. It will therefore be necessary to run a blank in duplicate on 1-gram samples of the salt as used. The tartaric acid recovered will represent the factor for obtaining the percentage of acid added from the weight of Rochelle salt used.

The procedure in the case of the blank consisted in merely diluting to 100 cc. and adding the acetic and other constituents, omitting, as above, the potassium acetate.

Total tartaric acid (grams per 100 cc.).

	Rochelle-salt method	Proposed method
No. 1	0.624	0.627
No. 2	1.170	1.205
No. 3	0.624	0.636
No. 4	0.620	0.638
No. 5	0.640	0.637

DISCUSSION OF RESULTS.

The general average of the results obtained by the collaborators for the two methods are:

Total tartaric acid (grams per 100 cc.).

	Provisional method	Proposed method
No. 1	0.554	0.622
No. 2	0.966	1.203
No. 3	0.538	0.618
No. 4	0.427	0.624
No. 5	0.576	0.619

With the exception of sample No. 2, the total tartaric-acid content of the five samples submitted is identical. Taking as a basis the general average (0.621) of the results obtained on these four samples by the proposed method, the recoveries by the provisional method are:

	Per cent
No. 1 (containing free tartaric acid)	89
No. 3 (containing 0.06 gram phosphoric acid)	87
No. 4 (containing 0.30 gram phosphoric acid)	70
No. 5 (containing no free tartaric acid)	93

Sample No. 2 had an addition of 0.569 gram of tartaric acid per 100 cc. Of this amount the proposed method recovered 102% and the provisional method 61%.

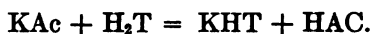
From these results it is evident that the provisional method is unreliable and inaccurate and that the proposed method gives very satisfactory results. Regarding the provisional method the results show—

(1) That it fails to determine all the free tartaric acid.

(2) That the addition of potassium acetate has a decomposing effect upon potassium-acid tartrate.

(3) That the presence of a free mineral acid such as phosphoric acid partially prevents the formation of potassium-acid tartrate.

The first two points, the presence or absence of free tartaric acid, are the two conditions occurring in natural wines and grape juices, whereas the presence of added phosphoric acid is very often met with in artificial grape products, such as grape-juice sirups and other soda-fountain beverages. Accordingly, the provisional method is not applicable to any of the conditions which occur in wines or grape juices, unless it is assumed that a relative amount of free tartaric acid retards the decomposing action of the potassium acetate upon potassium-acid tartrate:



In conclusion it should be said that the deductions made from the results obtained in this investigation are similar to those made in the three previous reports on this subject.

In making a recommendation for the adoption of the proposed method, it seems desirable to call attention to the fact that many results have been published on authentic samples of wines and grape juices of known purity examined by the provisional method. Results obtained by the proposed method would not, therefore, be comparable with previously published figures. It does not seem, however, that a method found to be entirely unsatisfactory should be retained by the association.

RECOMMENDATION.

It is recommended—

That the provisional method be dropped by the association and the proposed method be adopted for the determination of total tartaric acid in wines, grape juice, and soda-fountain sirups.

The following is the method for wines:

Neutralize 100 cc. wine with normal sodium hydroxid. The amount of alkali necessary for neutralization is calculated from the acidity of the wine. If the volume of the solution is increased more than 10% by the addition of the alkali, evaporate the solution to approximately 100 cc. Add tartaric acid to the neutralized solution. For each cubic centimeter normal alkali used add 0.075 gram tartaric acid.¹ After the tartaric acid has dissolved, add 2 cc. glacial acetic acid and 15 grams potassium chlorid. After the potassium chlorid has dissolved, add 15 cc. of 95% alcohol, stir vigorously until the cream of tartar starts to precipitate, and let stand for at least 15 hours in an ice box. Decant the liquid from the separated acid-potassium tartrate on a Gooch prepared with a very thin film of asbestos or on filter paper in a Büchner funnel. Wash the precipitate and filter three times with a small amount of mixture of 15 grams of potassium chlorid, 20 cc. of 95% alcohol, and 100 cc. of water, using not more than 20 cc. of the wash solution in all. Transfer the asbestos or paper and precipitate to the beaker in which the precipitation took place, wash out the Gooch or Büchner funnel with hot water, add about 50 cc. of hot water, heat to boiling, and titrate the hot solution with tenth normal sodium hydroxid, using phenolphthalein as indicator. Increase the number of cubic centimeters of N/10 alkali required by 1.5 cc. on account of solubility of the precipitate. One cubic centimeter of N/10 alkali is equivalent to 0.015 gram tartaric acid. To ascertain the tartaric acid originally present in the wine, subtract the amount of tartaric acid added. This will give the grams of total tartaric acid per 100 cc. of wine.

¹ The tartaric acid used should be of the purest obtainable and should be powdered and dried for about two hours at the temperature of boiling water.

In the case of grape juice or sirups, use 50 cc. of the sample, neutralize with normal sodium hydroxid, and add distilled water to 100 cc. volume. Proceed as under wine, adding 20 cc. of alcohol instead of 15 cc. After subtracting the tartaric acid added, multiply by 2 to obtain grams total tartaric acid in 100 cc.

REPORT ON BEER.

BY H. S. PAINE (Bureau of Chemistry, Washington, D. C.),
*Associate Referee.*¹

No plan for further work was submitted in the last report of the associate referee on this subject and, owing to a combination of circumstances, a plan for work which was deemed to be sufficiently profitable was devised too late for submittal to the collaborators.

Briefly, the matter may be stated as follows: Comparison of the analyses of various malt beverages, correlated with a knowledge of the conditions of the mash, leads to the conclusion that the methods employed for the determination of maltose (or "reducing sugars") and dextrin do not yield accurate results in all cases—the degree of accuracy being variable—that is, fairly acceptable in some cases and quite unsatisfactory in others. This variation is probably dependent upon the proportion of maltose and dextrin, and this in turn depends, of course, upon the mash conditions.

In the method which is based upon polarization there is a grave question of the accuracy of the assumed rotatory power of dextrin. There are various dextrans, of course, and it is a question whether the assumed value is always representative.

In the reduction method, which is more reliable, the factors which must be considered are somewhat as follows:

(1) The effect of dextrin and other constituents upon the reducing action of maltose in the reduction before hydrolysis, i.e., the question of applicability of the maltose tables to these conditions.

(2) The possibility of some reducing action by the lower dextrans.

(3) The possible effect of other sugars which might be present.

(4) The accuracy of the factors employed.

Various other considerations have a possible effect.

It is impossible in the absence of some preliminary experimental work to say just what the effect of these various factors may be and which will be of the most importance. The point to be emphasized, however, is that the variations in the composition of the mash and conditions of mashing are the controlling factors which cause a variable degree of accuracy in the above determinations. The other methods employed in beer analysis are, on the whole, satisfactory.

It is believed that the carbohydrate determinations are most in need of attention at the present time, and it is recommended that the methods for the determination of maltose and dextrin in beer be made the subject of study for the coming year.

No report was made by the associate referee on distilled liquors.

¹ Presented by M. J. Ingle.

A STUDY OF THE FUSEL OIL AND ESTERS IN
DISTILLED LIQUORS.BY LOUIS KATZ (Bureau of Chemistry Food and Drug Inspection
Laboratory, New York).¹

The examination of brandies and other distilled liquors for fusel oil is ordinarily limited to an estimation of the quantity of fusel oil present. No attempt is made to gain an insight into the composition of the fusel oil. Considering the importance of the fusel oil constituent of distilled liquors as a basis of judgment to be pronounced by the chemist as to their genuineness, and further considering the comparative ease with which the recognized minimum quantity of fusel oil could be added to neutral spirits, it seemed quite obvious that a mere quantitative determination of fusel oil is a somewhat weak link in the chain of evidence the chemist gathers in the examination of liquors. At best it enables the chemist to pronounce a liquor sophisticated only in that limited number of cases where not enough of a so-called bona-fier or too much neutral spirits have been added to keep the quantity of fusel oil above the required minimum. A qualitative examination of the fusel oil of distilled liquors, supplementary to its quantitative determination, seemed highly desirable and was accordingly undertaken at the suggestion of Mr. A. F. Seeker. The property of the volatile acids of the acetic acid series, when fractionally distilled from an aqueous solution, to pass over at a rate which is definite and characteristic for each acid,² was made the basis of the study. This property of the individual acids causes any definite mixture of them to distil over at a rate which is definite and characteristic for that particular acid mixture. In the regular course of analysis of distilled liquors, the so-called fusel oil, or the sum total of the higher alcohols, is determined quantitatively by oxidizing these alcohols to their corresponding acids and titrating the acid mixture with alkali. Now this acid mixture, after having been titrated and thus having served the purpose of quantitative estimation of the fusel oil, was again acidified with sulphuric acid and submitted to fractional distillation. As was expected, the rate of distillation differed in the case of acid mixtures obtained from the fusel oils of different samples of liquors, such differences being obviously due to differences in the composition of the acid mixtures, hence also of the fusel oils in these samples. In other words, the fusel oil of a distilled liquor yields upon oxidation an acid mixture which, when fractionally distilled from an aqueous solution under definite conditions, passes over at a rate which is dependent upon the composi-

¹ Presented by A. F. Seeker.² See method of M. Duclaux of detecting and estimating organic acids. Allen's Commercial Organic Analysis, 1905, 1: 490.

tion of the fusel oil. The curve representing that rate of distillation affords, therefore, a ready means to differentiate between fusel oils of different composition without making an actual analysis of those complex mixtures.

The esters of distilled liquors may also be studied qualitatively by isolating the sum total of their combined acid radicles and submitting the acid mixture to fractional distillation.

The procedure was in all cases as follows:

To 119 cc. of the acid mixture in a 500 cc. distillation flask, 1 cc. of concentrated H_2SO_4 was added, the flask connected to a condenser, and the solution distilled at a moderate rate, the receiver being changed each time 20 cc. passed over. Five fractions of 20 cc. each were distilled over, 20 cc. remaining in the distillation flask. Each fraction was titrated with standard alkali, thus furnishing a set of five numbers, representing the rate of distillation of the mixed acids, from which a curve graphically showing this rate of distillation could be estimated. The numbers were expressed as percentages of acid in each fraction with regard to the total acid that had passed over in the entire 100 cc. distilled. Thus, a rate of distillation represented by the following five numbers: 40, 25, 15, 10, 10, would mean that the first 20 cc. fraction contained 40% of total acid that distilled over in all the five fractions taken together, the second 20 cc. fraction contained only 25%, the third, 15%; the fourth, 10%, and the fifth, 10%.

Tables 1, 2, and 3 represent the results of fractional distillation of acid mixtures resulting from the oxidation of fusel oils derived from various distilled liquors.

TABLE 1.
Fractional distillation of fusel oil—French brandies.

SAMPLE NO.	PER CENT OF ACID IN EACH FRACTION					SAMPLE NO.	PER CENT OF ACID IN EACH FRACTION				
	1	2	3	4	5		1	2	3	4	5
1	41.0	25.9	15.6	9.6	7.8	20	36.2	23.2	15.2	12.4	13.0
2	41.0	24.8	15.4	10.2	8.5	21	38.6	23.9	14.5	11.0	12.0
3	40.6	22.6	15.1	10.9	10.8	22	38.2	23.1	15.6	11.6	11.5
4	40.7	23.2	13.9	11.1	11.1	23	40.6	24.1	14.4	10.7	10.2
5	43.8	21.9	14.4	9.6	10.3	24	41.9	23.9	14.4	10.5	9.3
6	38.3	23.4	17.0	10.7	10.6	25	38.0	23.0	15.6	11.8	11.6
7	42.4	24.3	14.4	10.8	8.1	26	38.3	24.0	15.3	11.2	11.2
8	40.2	23.9	15.2	10.9	9.8	27	40.1	22.9	14.4	10.6	11.9
9	42.7	24.3	15.5	8.8	8.7	28	40.1	23.2	15.0	10.6	11.1
10	45.7	23.1	13.0	9.2	8.9	29	41.2	22.4	13.7	10.7	12.0
11	39.4	24.8	15.2	10.4	10.2	30	40.3	24.3	14.8	10.4	10.2
12	38.9	23.6	15.4	10.9	11.2	31	41.8	24.6	15.6	9.8	8.2
13	41.1	24.3	14.8	10.0	9.8	32	41.9	25.0	14.5	10.2	8.4
14	41.5	22.9	14.6	10.6	10.4	33	38.5	24.8	13.7	11.2	11.8
15	45.5	23.5	13.4	9.0	8.6	34	36.0	23.6	15.6	12.4	12.4
16	46.6	23.3	13.5	8.2	8.4	35	45.2	25.2	14.0	8.3	7.3
17	36.0	23.4	15.1	12.0	13.5	36	41.2	24.3	15.0	10.5	9.0
18	38.0	23.2	15.1	11.4	12.3	37	39.1	24.2	15.2	11.4	10.1
19	38.4	23.5	14.4	11.6	12.1	38	37.3	23.7	15.9	11.9	11.2

Table 4 is a study, on identical lines, of the fractional distillation of acid mixtures representing the acid radicles of the esters. In the last case those acid mixtures were isolated in the following manner: The free acidity of the liquor having been determined in a separate portion, 100 cc. of it was neutralized exactly with the required amount of standard alkali and distilled. The distillate was saponified in the ordinary manner (as in the determination of esters) and again distilled till all alcohol had passed over. The residue in the distillation flask being then diluted with water, acidified with H_2SO_4 and distilled, the residue in the distillation

TABLE 2.
Fractional distillation of fusel oil—miscellaneous brandies.

SAMPLE NO.	PER CENT OF ACID IN EACH FRACTION					SAMPLE NO.	PER CENT OF ACID IN EACH FRACTION				
	1	2	3	4	5		1	2	3	4	5
French brandy						60	42.7	24.4	14.5	9.8	8.6
39	40.8	23.4	15.1	10.6	10.1	61	36.0	24.0	16.7	12.0	11.3
40	41.3	24.0	14.9	10.3	9.5	62	47.6	25.5	13.7	7.4	5.8
41	40.8	24.1	15.4	10.5	9.2	63	38.3	24.7	15.5	11.4	10.1
42	40.3	24.6	15.3	10.9	8.9	Hungarian brandy					
43	40.4	25.7	15.5	10.6	7.8	1	37.8	18.9	16.2	13.5	13.5
44	40.2	23.9	15.0	10.8	10.1	2	35.4	21.0	14.8	13.7	15.1
45	38.9	24.0	14.9	11.1	11.1	3	34.7	22.2	15.3	13.9	13.9
46	41.7	24.2	14.6	10.4	9.1	4	41.6	23.4	14.7	10.5	9.8
47	40.0	24.2	15.3	11.0	9.5	5	38.7	22.9	15.0	11.7	11.7
48	35.0	23.5	16.5	12.5	12.5	Italian brandy					
49	47.2	24.6	13.3	8.3	6.6	1	48.5	24.2	12.6	7.7	7.0
50	41.0	23.9	14.9	10.4	9.8	Greek brandy					
51	44.5	25.3	14.6	8.9	6.7	1	38.1	22.0	14.8	11.9	13.2
52	42.3	24.0	14.7	10.0	9.0	2	46.4	23.7	12.4	8.3	9.2
53	40.2	25.1	15.9	10.5	8.3	3	48.8	24.1	12.7	7.5	6.9
54	35.9	22.5	15.9	13.1	12.6	4	34.8	22.8	15.4	13.1	13.9
55	44.5	25.4	15.0	8.7	6.4	5	47.8	23.8	12.3	7.8	8.3
56	38.8	23.6	15.2	11.9	10.5	6	48.0	23.8	14.5	7.7	6.0
57	42.9	25.2	14.1	9.8	8.0						
58	37.9	24.2	15.5	11.8	10.6						
59	35.9	23.3	16.8	12.6	11.4						

flask was again diluted and again distilled. The combined distillates containing the volatile acid radicles of the esters were submitted to fractional distillation in the manner described above for fusel oil.

Of all the samples examined, only the eight samples marked "Authentic cognac" are known to be genuine cognacs. All the other samples are marked according to the legend on the label, and their history, origin, and mode of manufacture are not known. Handicapped as we are because of this uncertainty as to the origin and peculiarities in the methods of manufacture of almost all of the samples under examination, still it is possible to draw some general conclusions from a study of the tables submitted.

TABLE 3.
Fractional distillation of fusel oil—miscellaneous liquors.

SAMPLE	PER CENT OF ACID IN EACH FRACTION				
	1	2	3	4	5
Spanish brandy:					
No. 1.....	36.7	22.0	15.3	13.0	13.0
No. 2.....	40.4	22.9	15.3	10.7	10.7
Chile brandy.....	41.3	24.0	14.6	10.4	9.7
Batavia arrac.....	27.7	19.7	16.2	16.8	19.6
German plum brandy.....	36.3	23.1	15.6	12.5	12.5
Slivovits.....	41.3	23.6	14.8	10.7	9.6
Pomace brandy (French):					
No. 1.....	36.6	21.7	14.7	12.6	14.4
No. 2.....	29.7	20.4	15.9	15.3	18.7
No. 3.....	29.9	19.6	15.7	15.6	19.2
Pomace brandy (Italian).....	30.9	21.4	16.4	14.6	16.7
Jamaica rum.....	24.1	20.7	19.3	17.9	18.0
West Indies rum.....	46.9	24.1	12.7	8.2	8.1
St. Croix rum.....	47.0	23.5	12.8	8.4	8.3
Cuba rum.....	35.8	22.7	16.1	13.1	12.3
French rum.....	41.3	23.8	14.8	10.1	10.0
Raisin brandy:					
No. 1.....	46.6	25.7	13.5	8.1	6.1
No. 2.....	39.2	23.7	14.4	11.4	11.3
Brandy:					
No. 1.....	35.7	22.3	15.9	12.7	13.4
No. 2.....	29.4	19.6	16.3	15.7	18.9
No. 3.....	28.0	20.2	16.9	15.2	19.7
No. 4.....	46.4	25.4	13.6	8.2	6.4
Authentic cognac:					
No. 1.....	46.6	24.8	13.0	8.1	7.5
No. 2.....	44.3	23.1	13.5	9.4	9.7
No. 3.....	45.1	24.5	13.1	8.9	8.4
No. 4.....	49.7	25.2	12.4	7.0	5.7
No. 5.....	48.5	24.5	12.4	7.6	7.0
No. 6.....	46.6	24.4	13.0	8.4	7.6
No. 7.....	49.6	23.8	12.2	7.4	7.0
No. 8.....	45.2	24.1	13.7	8.5	8.5
Commercial fusel oil.....	49.9	24.6	12.0	7.0	6.5

(1) The combined acids of the esters in distilled liquors seem to be surprisingly constant in their composition, and their rate of distillation does not, therefore, offer as promising a means for detecting sophistication as is undoubtedly the case with the higher alcohols.

(2) In the case of the acids obtained by oxidation of the higher alcohols of brandies, the greatest variations are shown by the values for the first and fifth fractions, their averages being for—

	Per cent	Per cent
Authentic cognacs.....	47.0	7.7
French brandies.....	40.5	9.9
Pomace brandies.....	31.8	17.2

The values of the fourth fraction are usually not much different from those of the fifth fraction, and the variations in the values of the second and third fractions are rather small.

(3) The rate of fractional distillation seems to offer a promising means of differentiating between cognacs, ordinary brandies, and pomace brandies, and generally between distilled liquors where difference in geographical origin or mode of manufacture has affected the composition of the fusel oil complex.

TABLE 4.
Fractional distillation of esters—miscellaneous liquors.

SAMPLE	PER CENT OF ACID IN EACH FRACTION				
	1	2	3	4	5
French brandy:					
No. 1.....	18.3	16.1	18.0	20.7	27.0
No. 2.....	17.0	16.2	18.2	21.0	27.5
No. 3.....	21.2	15.9	17.1	20.5	25.2
No. 4.....	17.4	17.8	17.8	20.4	26.6
No. 5.....	16.7	16.8	18.3	21.3	26.9
No. 6.....	17.0	17.0	18.1	21.3	26.6
No. 7.....	18.1	16.1	18.0	20.6	27.2
No. 8.....	16.9	16.0	16.5	21.4	29.2
No. 9.....	16.6	16.2	18.2	21.2	27.8
No. 10.....	17.0	16.3	18.1	20.7	27.9
No. 11.....	18.8	16.3	18.0	19.8	27.1
No. 12.....	16.6	16.4	18.2	21.1	27.7
No. 13.....	17.6	16.3	17.2	21.1	27.8
No. 14.....	17.3	16.5	18.4	20.8	27.0
No. 15.....	17.3	16.9	18.5	21.3	26.0
No. 16.....	17.5	16.5	18.1	21.2	26.7
Authentic cognac:					
No. 1.....	21.0	17.3	17.3	19.4	25.0
No. 2.....	19.4	16.4	17.9	20.2	26.1
No. 3.....	20.3	17.0	18.0	19.8	24.9
No. 4.....	20.9	17.7	18.0	19.3	24.2
Pomace brandy:					
No. 1.....	19.4	17.0	17.6	20.7	25.3
No. 2.....	17.6	16.7	18.1	21.1	26.5
No. 3.....	19.0	16.6	18.6	20.0	25.8
Greek brandy:					
No. 1.....	23.4	16.7	17.5	18.5	23.9
No. 2.....	18.2	16.1	18.4	20.9	26.4
No. 3.....	16.0	16.0	18.0	20.9	29.1
No. 4.....	16.6	13.3	19.4	22.0	28.7
Rum (Jamaica).....	19.2	17.3	17.9	20.3	25.3
Rum (French):					
No. 1.....	20.4	15.8	17.3	20.6	25.9
No. 2.....	18.4	16.7	18.1	21.1	25.7
Rum (Martinique).....	19.6	16.7	18.1	19.2	26.4
Rum (St. Croix).....	16.4	16.4	17.5	21.2	28.5
Rum (West Indies).....	15.7	16.7	18.5	21.8	27.3
Apricot liquor.....	18.9	16.8	18.8	20.4	25.1
Batavia arrac.....	15.9	16.7	18.2	21.1	28.1

(4) In one case—that of French brandy (No. 17, Table 1)—which gave upon analysis rather high figures for fusel oil and esters, thus apparently indicating good quality, suspicion as to its genuineness was aroused by the fact that the fractional distillation of the acids from the fusel oil gave the following figures: 36.0, 23.4, 15.1, 12.1, 13.5, indicative of

pomace rather than of ordinary brandy. The sample was thereupon submitted to two expert tasters, both of whom agreed in pronouncing the sample sophisticated. There are a number of brandies marked "French brandies" in Tables 1 and 2 whose fractional distillation curves are more or less similar to the curve mentioned above, and thus point the same way; that is, that they are not genuine brandies.

(5) A comparative organoleptic test of the "Authentic cognacs" seems to point to the fact that the finer the flavor and aroma of the cognac the higher the value of the first fraction in the fractional distillation.

(6) To sum up: The results so far obtained warrant the belief that valuable information relative to the origin and character of distilled liquors can be gained by ascertaining the rate of distillation of the mixed acids obtained by oxidation of their higher alcohols. In order to establish the value and limitations of the method, it will be necessary to work upon a sufficiently large number of samples from various sources the history of which is well authenticated.

APPLICATION OF THE PROCEDURE TO THE ANALYSIS OF A FLAVORING ESSENCE.

Table 5 shows the rate of distillation of formic, acetic, propionic, butyric, and valeric acids and also of simple mixtures of some of these acids when distilled under conditions indicated above in the discussion of fusel oils in distilled liquors.

The figures in the table are averages of two determinations in each case except in the case of formic acid, where an average was taken of four determinations. The separate determinations ordinarily check within 0.5%, but formic acid behaves in a rather erratic manner, its figures varying sometimes as much as 2%. Column 6 of Table 5 gives the percentage relation of the total acid in the total distillate of 100 cc. to the acid originally present in the distillation flask.

It is obvious that use may be made of the fractional distillation for the following purposes:

- (1) Identification of any of the acids under discussion when present in a state of purity.
- (2) Isolation of any of the acids in a pure state from a mixture of two and sometimes more acids for the purpose of identification.
- (3) Testing for purity of any of the above acids.
- (4) Analysis of a simple mixture of two and sometimes even more of the acids.

The above is the basis of M. Duclaux's method (as quoted in Allen's *Commercial Organic Analysis*) of identification and estimation of the lower acids of the acetic acid series.¹ In this connection it is well to note that

¹ See also article by A. Landolt, *Unterscheidung natürllicher und künstlicher Fruchtäther*, in *Chem. Ztg.*, 1911, 35: 677.

the composition of an acid mixture cannot always be computed from its fractional distillation curve with any satisfactory degree of accuracy, as the acids mutually affect each other during distillation. The curve of the mixture nevertheless gives an approximate idea of its composition, and two or three experiments are generally sufficient to make up a known mixture of the given acids that will yield upon fractional distillation a curve practically identical with the one from the unknown mixture. The total acid in both the known and unknown mixture should be approximately the same in order that the results of their fractional dis-

TABLE 5.
Fractional distillation of acids.

ACID IN DISTILLATION FLASK	MIXTURES	PER CENT OF ACID IN EACH FRACTION					TOTAL ACID IN COMBINED DISTILLATE
		1	2	3	4	5	
cc. N/10							per cent
8.5-17.7	Formic acid.....	11.6	13.9	17.2	22.0	35.3	42.8
11.6-7.5	Acetic acid.....	14.8	17.1	18.9	21.8	27.4	68.1
6.4-12.8	Propionic acid.....	24.5	22.6	20.7	17.8	14.4	92.1
7.3-15.6	Butyric acid ¹	36.1	27.1	18.4	11.8	6.6	94.0
11	Valeric acid ²	54.6	27.2	12.2	4.3	1.7	97.0
30	2 molecules valeric plus 1 molecule acetic.....	45.6	24.8	13.5	8.5	7.6	87.5
21	2 molecules valeric plus 1 molecule propionic..	44.7	25.9	15.0	8.8	5.6	96
21	2 molecules valeric plus ½ molecule acetic plus ½ molecule formic.....	46.5	24.8	13.4	8.1	7.1	84
58	1 molecule valeric (14.5 cc.) plus 3 molecules acetic (43.5 cc.).....	28.2	20.5	16.8	16.1	18.4
.....	1 molecule valeric (11.6 cc.) plus 4 molecules acetic (46.4 cc.).....	25.8	20.0	17.2	17.1	19.9
48	1 molecule valeric (8 cc.) plus 5 molecules acetic (40 cc.).....	24.4	19.4	17.4	17.7	21.1
49	1 molecule valeric (7 cc.) plus 6 molecules acetic (42 cc.).....	23.0	19.2	17.6	18.4	21.8

¹ Of uncertain purity.

² Obtained by oxidation of amyl alcohol (Kahlbaum's).

tillation might be comparable, as the fractional distillation curve is somewhat affected by the concentration of the acid to be distilled. The limits of permissible variation in the strength of the acid to be distilled without affecting the distillation curve have not been determined, though, as will be seen from Table 5, a 100% variation in the strength of an approximately N/100 acid does not materially affect its distillation curve.

A complete analysis of a flavoring essence—a so-called banana oil—has been made in this laboratory by a procedure based largely on the above-described facts and considerations involving the fractional dis-

tillation of acids and their mixtures. A detailed description of the procedure followed will now be given as an illustration of the possibilities of the method.

DESCRIPTION OF METHOD.

About 5 grams of the banana oil was saponified with an excess of aqueous KOH by boiling under reflux condenser. The total alcohols in the saponification product were then distilled off and thus two solutions obtained:

(1) The residue in the distillation flask containing the total combined acids from the esters, together with a small amount of acid originally free in the oil and which has been determined in a separate portion of the oil by direct titration; and

(2) The alcoholic distillate containing the total alcohol, free and combined, of the original oil.

The excess of alkali in solution (1) was titrated with sulphuric acid and the total combined and free acid in the oil thus estimated. The neutralized solution was then acidified and an aliquot fractionally distilled. The values obtained for the fractions were as follows: 22.7, 19.1, 17.7, 18.8, 21.7. Ratio of acid in total distillate to acid in distillation flask, 72%.

With a view of isolating the component acids of the mixture, fraction 1 and also the residue in the distillation flask were, upon proper dilution, again fractionally distilled. The latter (the residue in distillation flask) yielded values for the fractions practically identical with those of pure acetic acid. The first fraction, however, yielded a curve indicating a mixture and not a simple acid. The first fraction in this, the second distillation, was again refractionated and the operation repeated until after the fifth fractional distillation of the first fractions values were obtained practically identical with those of a pure valeric acid. Thus was the acid mixture in the saponification product proved to be a mixture of valeric and acetic acids. It was then established by direct experiment that a mixture of one molecule valeric and six molecules acetic acids would yield upon fractional distillation the following values: 23.0, 19.2, 17.6, 18.4, 21.8. Ratio of acids in total distillate to acid in distillation flask, 71.7%.

The above values are practically identical with those obtained from the acid mixture in the saponification product of the banana oil. Thus was the composition of the total acid (free and combined) in the banana oil determined to be as follows:

	cc. N/10 per gram
Free acid (assumed to be acetic).....	2.0
Total combined acids (by saponification).....	67.4
Combined acetic acid (by fractional distillation).....	57.5
Combined valeric acid (by fractional distillation).....	9.9

An aliquot of the solution of the total acids was neutralized, evaporated off, dried, and weighed, and the weight agreed fairly well with the composition indicated above.

The alcoholic distillate from the saponification product containing the total alcohol (free and combined as esters) of the banana oil was oxidized with alkaline permanganate to their corresponding acids.¹ The resulting acids were distilled off, the total acidity of the distillate titrated, and the solution then examined for its com-

¹See method of C. R. Smith and A. S. Mitchell, U. S. Bur. Chem. Bul. 122: "Determination of Fusel Oil by Alkaline Permanganate."

ponent acids qualitatively and quantitatively by a system of fractional distillation, exactly in the same manner as outlined above for the combined and free acids in the oil.

The acid mixture resulting from the oxidation of the total alcohols (free and combined) in the oil proved to be composed as follows:

	cc. N/10 per gram
Valeric acid.....	55
Acetic acid.....	30

thus indicating that the total alcohols were composed as follows:

	cc. N/10 per gram
Amyl alcohol.....	55
Ethyl alcohol.....	30

The above results were checked in the following manner:

The original banana oil was saponified with alcoholic potash, all the alcohol in the saponification product distilled off, and the alcoholic distillate examined for higher alcohols in exactly the same manner as fusel oil is determined in brandy (Allan Marquart fusel-oil method). Titration of the final acid distillate gave 55 cc. N/10 acid per 1 gram oil, and the fractional distillation of the solution gave values for the fractions identical with those of pure valeric acid.

Further examination of the banana oil gave the following data:

Free ethyl alcohol was proved to be absent; the presence of amyl acetate, ethyl acetate, and amyl valerate was indicated by the odor of various fractions obtained from a fractional distillation of the original banana oil; nonvolatile unsaponifiable matter, 1%.

Summing up the results obtained, we arrive at the following composition:

	cc. N/10 per gram
Free acid (as acetic).....	2.0
Combined acetic acid.....	57.5
Combined valeric acid.....	9.9
Combined ethyl alcohol.....	30.0
Combined amyl alcohol.....	37.4
Free amyl alcohol.....	17.6
Nonvolatile unsaponifiable (per cent).....	1

and the rational formula may be expressed as follows:

	cc. N/10 per gram	per cent
Free acid (as acetic).....	2.0	1.2
Amyl valerate.....	9.9	17.0
Ethyl acetate.....	30.0	28.4
Amyl acetate.....	27.5	35.7
Free amyl alcohol.....	17.6	15.5
Nonvolatile unsaponifiable.....		1.0
		<hr/> 96.8

No report was made by the associate referee on vinegar.

REPORT ON SPICES.

By HARRY E. SINDALL (Philadelphia, Pa.), *Associate Referee.*

The work done this year falls under two headings—moisture determinations on whole spices and the total ash of herbs.

MOISTURE.

No recommendations were made by last year's associate referee for further work on spices, but it seemed desirable this year to study a method for determining moisture in whole spices. No samples were sent out, as the nature of the work rendered it unsuitable for coöperative investigation. The work was done on whole cloves and whole black pepper. The method under consideration was the Brown-Duvel distillation method for moisture in grain. But since unsatisfactory results were obtained by using Renown engine oil, or in fact any engine oil, the best grade kerosene oil was substituted and found to give more uniform results.

The method as worked by the associate referee is as follows:

Place 50 grams of whole spice in a distilling flask with 150 cc. kerosene, whirl the flask several times to bring the oil in contact with each particle of spice. Place the flask on an asbestos board. Cut so that the bottom of the flask extends below the surface. A wire gauze with an asbestos center is placed about one-half of an inch below the bottom of the flask. The object is not to bring the flame in direct contact with the flask, and the asbestos board serves to keep the heat uniform. Connect the flask directly with a vertical condenser and collect the distillate in a graduated cylinder or burette. Insert a thermometer through the stopper of the distilling flask, extending down into the oil. Adjust the flame so that about twenty minutes will be required to reach the temperature of 170°C. Now extinguish the flame, after which the thermometer will show a slight gradual increase in temperature. As soon as the water stops dropping from the condenser tube, which usually requires from four to six minutes, the operation is complete. Multiply the volume of the water layer by 2 to obtain the percentage of moisture.

The following table shows results obtained by drying 2 grams of whole cloves and whole black pepper at 110° C. to constant weight; from the resulting loss in weight the amount of volatile ether extract obtained by extracting the whole spices was subtracted.

The samples marked (a) were taken before the cloves went on the mill to be ground; the samples marked (b) represent the same cloves immediately after grinding. The loss according to weight by grinding was 6.26%.

Comparative results on moisture.

BY EXTRACTION		BY DISTILLATION		BY EXTRACTION		BY DISTILLATION	
	per cent		per cent		per cent		per cent
Whole cloves ..	(a) 13.94	(a) 11.2	Ground cloves..	10.16		
Do.....	15.98	12.6	Whole pepper..	6.02		6.0	
Do.....	11.24	9.8	Do.....	7.95		6.8	
Do.....	10.61	9.5	Ground pepper.	7.87		8.0	
Ground cloves.	(b) 6.11	(b) 6.0					

This loss consists chiefly of moisture and some volatile oil, due to the fact that the cloves are more or less heated while passing through a high-speed mill.

The large percentage of moisture in the cloves as shown in the above table under extraction method seems due to the fact that all the volatile oil was not extracted by the ether, as shown in the following table:

Volatile ether extract.

	<i>per cent</i>		<i>per cent</i>
Whole cloves.....	2.88	Ground cloves.....	15.74
Do.....	2.89	Whole pepper.....	0.30
Do.....	3.13	Do.....	0.53
Do.....	3.31	Ground pepper.....	2.13
Ground cloves.....	13.91		

TOTAL ASH DETERMINATION.

The associate referee's attention was called to wide discrepancies in the results of total ash determinations obtained by different chemists on samples taken from the same lot of herbs. For the purpose of studying this matter, large samples of marjoram, sage, savory, and thyme were ground under the associate referee's supervision, and subdivisions of these were sent to collaborators, with instructions to determine the total ash in each sample by the method commonly used by the respective collaborators and describe the method used. Reports were received from eight collaborators:

Total ash.

ANALYST	MARJORAM	SAGE	SAVORY	THYME
	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>
F. L. Shannon, Lansing, Mich....	{ (a) 14.34	(a) 9.265	(a) 13.335	(a) 12.89
	{ (b) 14.135	(b) 9.275	(b) 13.29	(b) 12.78
C. L. Black, Philadelphia, Pa....	16.16	9.45	14.06	13.14
H. B. Mead, Philadelphia, Pa....	15.84	9.75	14.05	13.42
C. S. Brinton, Philadelphia, Pa..	{ 15.21	9.39	13.63	13.47
	{ 15.10	9.43	13.72	13.21
A. P. Coulture, Ottawa, Canada..	{ 14.54	9.47	13.68	12.47
	{ 14.61	9.50	13.56	12.59
Paul Rudnick, Chicago, Ill.....	{ 13.42	9.31	12.60	13.14
	{ 13.44	11.21	12.68	12.48
C. O. Dodge, Washington, D. C...	14.05	8.41	12.41	13.46
J. H. Bornmann, Chicago, Ill....	{ 14.08	9.80	13.77	13.67
	{ 14.48	9.82	13.85	13.43

Mr. Dodge also determined nonvolatile matter and ash on a nonvolatile basis, with the following results:

	NONVOLATILE MATTER	ASH ON NON- VOLATILE BASIS
	<i>per cent</i>	<i>per cent</i>
Marjoram.....	92.6	15.17
Sage.....	93.0	9.05
Savory.....	91.6	13.54
Thyme.....	92.9	14.48

METHODS OF COLLABORATORS.

Mr. Shannon made the determinations in duplicate. Sample (a) in each case was ignited in an ordinary platinum milk dish; sample (b) in a fused silica dish of the same size. All were burned first over a Meker burner at a very low red heat and then exposed to a low red heat in a muffle, cooled in a desiccator, and weighed.

Mr. Black used 2-gram portions in platinum dishes which were held about one-fourth inch above bottom of muffle by means of asbestos board supported by small pieces of pipestem. The bottom of the muffle was kept at a barely visible red heat. After ashing was apparently complete, the residue was moistened with alcohol to reveal any unburned carbon and then reheated if necessary until treatment showed no unburned carbon.

Mr. Mead used 2-gram samples in flat-bottom round dishes, dimensions about 3 inches by 1 inch. These were elevated on pieces of pipestems about three-eighths inch above the bottom of the gas muffle heated to a visible red. The dishes were below visible red. The ash was moistened with alcohol and reheated. There was practically no carbon or change after the first heating.

Mr. Brinton used 2-gram portions and made the combustion in small platinum dishes about 1 inch square and approximately three-eighths inch deep. At no time during any of the work were the dishes heated to a red heat. The determinations were made in a gas-heated muffle, the bottom of the muffle at times being more or less red; but the dishes were supported on pieces of asbestos board about one-fourth inch or more above the bottom of the muffle. An air space, therefore, separated the asbestos board from the red-hot bottom of the muffle. The asbestos board was supported by pieces of crucible lid.

Mr. Coulture used 2-gram samples, and charred them in platinum at a heat below redness. This temperature was found to be sufficient to completely burn off all the carbon. He did not use a muffle. After weighing, dish and contents were heated again slightly below red heat for a period of half an hour and the operation repeated to constant weight. After a time there was a slight increase in weight, and the minimum was taken as being the true weight of the ash.

Mr. Rudnick used 2 grams in a small flat-bottom porcelain dish and ignited carefully at a low temperature so as to avoid loss of sample. The ignition was continued at a low red heat and finally at a bright red for a few minutes, then cooled and weighed. The appearance of the ash after weighing was carefully noted to be sure that all carbon had been burned off, also to note whether particles of sand and foreign matter other than true ash were apparent.

Mr. Dodge followed the method given in U. S. Bureau of Chemistry Bulletin 107 (revised), except that the products were dried at 110°C. for four hours and weighed, then dried for one hour and again weighed. The greatest loss during the second drying was less than 1%.

Mr. Bornmann charred 2 grams of the well-mixed substance in a platinum dish over asbestos, using a small flame. The carbon was burned off in a muffle at a temperature below redness.

CONCLUSIONS.

These results are not encouraging. While the samples submitted were known to contain several per cent of sand, it is not believed that the discrepancies can possibly be due entirely to this cause, but that they were attributable, in part at least, to the temperature employed in mak-

ing the combustion. It would seem, at all events, that the subject warrants further investigation, particularly along the line of operating at definite temperatures.

RECOMMENDATIONS.

It is recommended—

(1) That the associate referee's modification of the distillation method for water in spices be given further study.

(2) That the subject of ash determination in herbs be further studied, with particular reference to the influence of the exact temperature employed in the combustion.

NOTE REGARDING THE DETERMINATION OF CRUDE FIBER IN BLACK PEPPER.

By A. E. PAUL (Bureau of Chemistry Food and Drug Inspection Laboratory, Chicago, Ill.).

Two samples, one with a low fiber content and the other high in this constituent, were sent to various laboratories in this district, with the request that analyses be made. The results follow:

Analyses of black pepper.

SAMPLE NO. 1.

ANALYST	NON-VOLATILE ETHER EXTRACT	VOLATILE ETHER EXTRACT	TOTAL ASH	ASH IN- SOLUBLE IN HCl	CRUDE FIBER	PENTO- SANS
	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>
J. H. Bornmann, Chicago... {	8.03	0.70	5.09	0.57	10.46
	8.27	0.83	5.41	0.61	11.38
E. H. Berry, Chicago.....	11.09
J. Feldbaum, Chicago.....	8.10	1.04	5.35	0.62	11.08
H. D. Grigsby, Cincinnati....	8.19	0.55	5.24	0.51	11.99
J. S. McCune, St. Louis.....	8.87	5.16	0.43	10.41	6.1
C. L. Clay, New Orleans.....	8.69	4.62	0.34	11.26	6.0

SAMPLE NO. 2.

	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>
J. H. Bornmann, Chicago... {	7.20	1.03	6.48	0.95	16.60
	7.26	1.01	6.61	1.00	17.11
E. H. Berry, Chicago.....	16.60
J. Feldbaum, Chicago.....	7.15	1.18	6.48	0.92	16.80
H. D. Grigsby, Cincinnati....	7.18	0.57	6.43	0.79	17.41
J. S. McCune, St. Louis.....	7.82	6.37	0.76	15.84	8.40
C. L. Clay, New Orleans.....	7.80	5.48	0.56	17.23	7.90

In view of the very great discrepancy in the various fiber determinations, a new 5-pound sample was taken by an analyst, carefully mixed, quartered to a small bulk, and transferred to a small sample bottle.

The remainder was again carefully mixed, and a small subsample withdrawn as before. Then still a third sample was taken in the same manner. The analyst was requested to make crude-fiber determinations in duplicate on each subdivision.

The remainder of the pepper was then delivered to another analyst, who prepared three subsamples in the same manner as above.

Both analysts were instructed to stir the material in the bottle very carefully with a spoon of approximately 2-grams capacity, to withdraw a spoonful from the interior of the bottle, and carefully transfer the entire contents to the balance pans. It was then left to the option of the analysts to utilize the quantity thus taken for the fiber determination, or else to remove carefully the slight excess over 2 grams, or add sufficient to make that amount. Of the two analysts, Mr. Berry preferred the former detail, while Mr. Bornmann chose to work on the exact 2 grams. Their results were:

ANALYST	SAMPLE NO. 1	SAMPLE NO. 2	SAMPLE NO. 3
	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>
E. H. Berry.....	13.36	12.98	13.46
	13.15	13.00	13.16
J. H. Bornmann.....	13.03	13.15	13.25
	13.25	13.07	13.31

These results are quite acceptable, and show that the trouble is not in the method itself, but rather in the manner of mixing and weighing out the samples.

PRELIMINARY REPORT ON DETERMINATION OF ASH, ESPECIALLY IN SPICES.

By CLEMENT S. BRINTON (Food and Drug Inspection Laboratory, U. S.
Appraiser's Stores, Philadelphia, Pa.)

The object of this paper is to emphasize the necessity of carefully following the directions for the determination of ash as given in the Official and Provisional Methods of Analysis of this association (U. S. Bur. Chem. Bul. 107, rev.), particularly where, under "Ash, official" for the analysis of foods and feeding stuffs, the following language is used: "* * * burn until free of carbon at the lowest possible heat" (p. 38). Again, under "Preparation of ash," etc., (p. 238), it is directed to "Conduct the combustion at a comparatively low temperature, never employing a full red heat * * *." Other similar directions could be cited.

The desirability of preparing the material mentioned herein arose through trouble in obtaining closely agreeing results for ash by different

analysts during the examination of spices, especially those having normally a high sand-free ash, such as marjoram with 11 to 16% of ash and thyme with from 10 to 14%.

This lack of agreement was found to be due not to faulty sampling but to lack of uniformity in the analytical method. This was ascertained by having portions of a large sample of marjoram finely ground, about 60 mesh, thoroughly mixed, and analyzed in seven laboratories in the Eastern States according to the procedure usually employed in those laboratories. Determinations were made in platinum dishes in all cases except laboratory 6, where a porcelain crucible was used. Electric muffles were used in laboratories 1 and 4, and probably in laboratory 5; gas muffles were used in laboratories 2, 3, and 7; and laboratory 6 burned in open flame. The results submitted are given in Table 1.

TABLE 1.
Ash determinations on marjoram—first report by collaborators.

LABORATORY	ANALYST	TOTAL ASH	REMARKS
		<i>per cent</i>	
1	W. C. T.....	15.21	{ Muffle below redness; time of burning, 1 hour. Not observed carefully to ascertain when free of carbon. Time of burning, 4 hours additional; otherwise same as above.
		15.24	
		15.16	
		15.18	
2	W. J. McG.....	13.47	{ Muffle high red; time of burning not stated. Heated to constant weight.
		13.45	
3	J. B. L.....	15.14	{ Muffle low cherry red; time of burning not stated. Ash stirred with rod from time to time until carbon burned. Analyst J. E. heated longer and found no loss in weight.
	J. E.....	14.90	
4	F. O. W.....	15.11	{ Muffle below redness; time of burning not stated. Moistened with water to observe presence of carbon; again heated and weighed when free of carbon.
		15.13	
		15.11	
		15.09	
5	L. P.....	15.40	{ Muffle below redness; time of burning not stated. Moistened with water after weighing and found free of carbon.
		15.30	
6	H. E. S.....	13.24	{ Open flame with crucible bright red; time of burning, 40 minutes. Ash determined as he usually does in spices.
		13.35	
7	C. L. B.....	15.68	{ Dish not visibly red; time of burning not stated. Ash treated with water after weighing to test for carbon.
		15.70	
	H. B. M.....	16.11	{ Bottom of muffle below visible red heat; time of burning not stated. Moistened and reheated before weighing; weighed after free of carbon.
		16.16	
		16.03	
		15.69	

The writer obtained results ranging from a maximum of 15.8% down to a minimum of 14.89%, according to conditions, all results being obtained when the dish was not at a visible red heat as viewed in the open muffle by daylight. All determinations were made in platinum dishes about 1 inch square, one-half inch deep, in a 5-burner gas Wiesnegg muffle. The results are given in Table 2.

TABLE 2.
Ash determinations on marjoram—results by author.

DATE (1915)	TOTAL ASH		REMARKS
	Dish 1	Dish 2	
	<i>per cent</i>	<i>per cent</i>	
May 24	15.43	15.46	Time of burning, 1½ hours in all. Dishes on bottom of muffle entire time. Heated 1½ hours below visible red heat, then raised to dull red for one-fourth hour.
	15.01	14.89	Muffle just visible red on bottom; time of burning, 35 minutes. Dishes on bottom of muffle entire time and carbon-free when moistened with alcohol.
May 25	15.37	15.75	Not a visible red; time of burning, 35 minutes. Both dishes about one-fourth inch above bottom of muffle on crucible lids. Muffle open. Dish with high result in front of other.
May 26	15.74	15.86	Not a visible red; time of burning, about 2½ hours. These results from same conditions as above except muffle not so hot. After 1½ hours tested but not carbon-free. After weighing and getting these results found carbon free.
June 3	15.06	15.39	Bottom dull red in spots; time of burning, about 2 hours. One dish in front of other; otherwise same as second set May 24. Dishes raised off bottom and not a visible red.
	15.20	15.48	Bottom dull red in spots; time of burning, only 35 minutes in all. Carbon all burned out. One dish in front of other. Dishes raised off bottom.
June 8	15.18	15.10	Bottom dull red in spots; time of burning, only 37 minutes in all. Carbon all burned out. Dishes side by side and supported one-fourth inch above bottom on asbestos board. Neither asbestos board nor dishes a visible red. Hot air could circulate under and over asbestos and dishes.
June 9	15.70	15.65	Time of burning, about 6 hours. Conditions same as on June 8, but lower temperature used. Both ashes moistened after weighing and free of carbon. Dishes side by side.

TABLE 2.—Continued.

DATE (1915)	TOTAL ASH		REMARKS
	Dish 1	Dish 2	
June 9	<i>per cent</i> 23.35	<i>per cent</i> 23.87	Muffle slightly red in spots; time of burning, 17 minutes. Much carbon present. Conditions same as preceding.
	16.18	16.20	Muffle slightly red in spots; time of burning, 10 minutes long. Carbon still present in both.
	15.88	15.68	Dishes not visible red; time of burning, additional 10 minutes. Traces of carbon in both; most in higher result.
	¹ 16.37	¹ 16.10	Dishes not visible red. Both moistened, evaporated to dryness, heated in muffle below redness before weighing.
	16.15	15.93	Dishes not visible red. Same as preceding after one-half hour longer in muffle.
	15.75	15.65	Dishes not visible red. Same as preceding after 1 hour longer in muffle; temperature slightly higher.

¹ Note the effect of moistening with water and subsequent long heating required to drive off this water and bring result down to that obtained before moistening.

The results reported on May 26 agreed closely with those obtained by Analyst C. L. B., but are slightly below those reported by Analyst H. B. M. Further attempts to duplicate the latter's results were in vain, as shown by work subsequent to May 26. This later work brought out very emphatically the fact that this sample of marjoram contained mineral matter very susceptible to slight changes in temperature, length of time of burning, etc., and that without exact duplication of temperature and other conditions closely agreeing results at different times could not be determined. See various results in Table 2 on June 3, June 8, June 9, and June 10, where determinations in duplicate in every particular, except that one dish was in front of the other and in slightly cooler part of muffle, show variations of 0.3%, and yet both are free of carbon.

It was found that only when the two dishes were placed side by side could close duplicates be obtained, as shown by results of June 8 and June 9, each set of which agrees closely; but there is a marked difference of about 0.5% because of different temperatures, the dishes in neither set being a visible red, although the bottom of muffle was dull red in spots. The second set of results on June 9 were made at a low temperature and weighed at short intervals to determine if some easily volatile mineral matter was present which was completely volatilized about the time the carbon was completely burned. The tabulated results show that lower results were obtained than reported by Analyst H. B. M., yet some carbon was still present. Just at this point, the writer learned that Analyst H. B. M. had moistened his ash before weighing to uncover unburned car-

bon, then added enough alcohol to make a mixture which would burn, ignited the alcohol and burned it off, and replaced the dish in muffle for a short time, then cooled and weighed as usual. Because of this analyst obtaining higher results than anyone else and higher than the writer could obtain in any other way, the last set in Table 2 when about free of carbon were moistened, treated with alcohol, etc., about the same as did Analyst H. B. M., and it was found that his high results as originally reported could be duplicated, but in no other way; therefore, the conclusion was made that the water treatment before weighing explained his higher results. Later work by the author confirms this conclusion (see Table 4). The work reported in Table 2 convinced the author that for marjoram especially, and probably in other substances high in ash, an accurate determination of the ash required that more care be given to the control of the temperature than is usually given, and it was thought that higher results would be reported by the various analysts if their attention was called to this point. Consequently, the following method was sent to the previous collaborators:

Take 2 grams of the well-mixed sample in a platinum dish and burn at *lowest possible temperature*, below a visible red heat, until free from carbon when examined by the eye; cool, and weigh as usual. Now moisten ash with 95% alcohol, and examine for small specks of carbon. If specks of carbon are found, ignite alcohol, allow to burn off, and then return dish to muffle and allow to heat a little longer at same temperature. Cool and weigh again, repeating this operation until all carbon is burned off.

All results obtained were requested, also an opinion as to the true value for ash in the sample of marjoram sent out, the various results previously submitted by all of them being sent also so they would see the reason for further work. The second set of results are given in Table 3.

TABLE 3.

Ash determinations on marjoram—second report by collaborators.

LABORATORY	ANALYST	TOTAL ASH		REMARKS
		Dish 1	Dish 2	
		<i>per cent</i>	<i>per cent</i>	
1	H. R. S. ¹	15.47	Time of burning not given. Two grams in flat-bottom platinum dish, center cold electric muffle; rheostat set for temperature below red heat and burned till free of carbon.
		15.56	Same as above, except that when entirely charred dish was withdrawn. Ash covered with alcohol, ignited, and again heated in muffle <i>below red heat</i> . After 2 treatments with alcohol carbon-free.

¹ General comment by analyst; "It is evident that prolonged heating at even very low temperature does cause a loss of inorganic matter by volatilization. Also the most practical and reliable method for determining ash in organic products is that outlined in method 3 above."

² NOTE.—No report was received from laboratories 6 and 7.

TABLE 3.—Continued.

LABORATORY	ANALYST	TOTAL ASH		REMARKS
		Dish 1	Dish 2	
		<i>per cent</i>	<i>per cent</i>	
1	H. R. S.....	16.00	Same as above at start. When charred, removed, cooled, and all soluble ash leached out with hot water and filtered in usual manner. Filter and char burned in dish below red heat. Solution of soluble ash added, evaporated to dryness on steam bath, and finally heated at low temperature and weighed.
		15.04	Same as above at start. Rheostat set for lowest possible temperature; allowed to heat all night (16 hours).
2	W. J. McG..	15.10	Time of burning not given. Heated in gas muffle just below red heat till carbon had disappeared, and after one alcohol treatment. After heating at red heat for 30 minutes, obtained 13.03%, 13.06%, and a further drop to 12.46%, 12.54% after 30 minutes more at red heat.
		15.18		
		15.24		
		14.96		
3	J. F. D.....	16.16	16.46	Visible carbon present.
		15.74	15.87	Do.
		15.63	15.77	Do.
		15.37	15.76	Visible carbon absent.
		15.36	15.77	Total time of burning, 9 hours. Constant weight. Six treatments with alcohol were required. Muffle at all times below visible red heat. These results are about 0.5% higher than previously reported from this laboratory, but analyst does not consider increase in accuracy justifies increase of time.
4	F. O. W....	15.03	Time of burning not given. "The heating was done very slowly at a low temperature with proper precautions until no specks of carbon were left."
		15.06		
		14.95		
5	L. P.....	15.66	15.65	Time of burning not given. Burned in electric muffle at lowest temperature possible. When carbon seemed all gone, weighed, then moistened with alcohol and found carbon-free. Temperature is slightly lower than used for first results.
		14.91	Time of burning not given. Obtained at higher temperature than above, but still a temperature which shows no redness in muffle or in dish.
		14.69	Time of burning not given. Obtained on another determination by higher heat than preceding. Analyst considers last two results worthless.

Comments on Table 3 by author: All laboratories submitting reports obtained higher results by using lower temperatures, etc., than first reported, with the single

exception of laboratory 4. Further, these higher results, with two exceptions, agree very well with those obtained in laboratory 7, and those of the author as given in Table 2. The results of Analyst H. R. S., where he used water to moisten ash, confirm those of Analyst H. B. M. and the author, where water was added. The tenacious combination of this water is remarkable since, as shown by the author in Table 2, about $1\frac{1}{2}$ hours' heating at approximately 400°C . was required to drive it off and bring the ash down to the same value as before its addition.

The author, about this time, suggested similar work to the associate referee on spices. He has already submitted his results.

TABLE 4.

Ash determinations on marjoram and stramonium with temperature control.

MARJORAM.

DATE (1915)	TOTAL ASH	MAXIMUM TEMPERATURE BY PYROMETER	TIME BURNING	ASH AFTER MOISTENING AND REHEATING	TIME REHEATING	MAXIMUM TEMPERATURE WHILE REHEATING	INCREASE DUE TO MOISTENING	REMARKS
	per cent	$^{\circ}\text{C}$	h. m.	per cent	h. m.	$^{\circ}\text{C}$.	per cent	
Oct. 8 ...	15.69	(¹)	(¹)	15.94	1	400	0.25	Muffle entirely closed except 1-inch hole in front and three-eighths inch hole in back. Do.
	15.90	400	(²)	16.20	1	400	0.30	
Oct. 12 ..	15.25	(²)	1 35	15.43	1 51	400	0.18	Quartz dish. Muffle wide open. An increase of about 0.2 % after almost 2 hours heating.

STRAMONIUM.

	per cent	$^{\circ}\text{C}$.	h. m.	per cent	h. m.	$^{\circ}\text{C}$.	per cent
Oct. 26 ..	18.21	400-440	4 30	18.45	1 50	400	0.24
	18.27	400-440	4 30	18.37	1 50	400	0.10

¹ Not noted.

² 440°C . during combustion of carbon, then constant at 400°C .

The data given in Table 4 show results obtained where a pyrometer was used in the open gas muffle. The majority of these results are not satisfactory because of difficulties in maintaining a constant temperature in the muffle. The temperature and results reported are only approximate, but show very plainly that where an accurate result is desired on a substance like marjoram, great care must be observed to have exactly identical conditions, or considerable variations must be expected. The effect of moistening the ash is again plainly brought out in this table.

The drug stramonium has also a high ash, and some little work has been done on one sample (see Table 4).

This paper is a preliminary one intended to emphasize certain phases of the determination of ash in spices, especially the necessity of using a low temperature, *below redness at lowest possible temperature*, as specified in the Official and Provisional Methods of Analysis (p. 162) previously cited. The writer expects to present more data later, together with conclusions as to the composition of the mineral matter which volatilizes so easily at or below redness. Evidence will also be presented to show that with products like marjoram, thyme, etc., it is better to determine ash in an open muffle, allowing plenty of circulation of air, instead of in a closed muffle, where, owing to an absence of oxygen, a longer time is required to burn the carbon.

CONCLUSIONS.

(1) The mineral matter in marjoram, thyme, and other plant products having a high ash is easily volatile to a very marked degree at a temperature just below redness.

(2) Great care is necessary by analysts in ashing these products. Temperatures below visible redness must be used or erroneous and low results will be obtained.

(3) After moistening the ash with water the weight before moistening is not regained at the temperature of combustion, even after a reheating of over one hour.

(4) Further work on this subject is necessary so that the official methods will plainly caution analysts against the troubles mentioned above.

The committee for the suggestion of changes in the constitution and by-laws, previously authorized by the association, was announced as follows: B. B. Ross, chairman; C. L. Alsberg, and H. D. Haskins.

REPORT ON FLAVORING EXTRACTS.

By A. E. PAUL (Bureau of Chemistry Food and Drug Inspection Laboratory, Chicago, Ill.), *Associate Referee*.¹

Three methods were studied last year by collaborators on flavoring extracts:

(1) The saponification method for wintergreen extract, devised by Hortvet and West.

(2) The brine method for anise and nutmeg extracts, as described by Hortvet and West.

(3) The Howard-Mitchell method, slightly modified, for peppermint, spearmint, and wintergreen extracts.

¹ Presented by E. K. Nelson.

In each case the method was recommended for further study by the association. Inasmuch as the results reported last year appeared to be quite satisfactory, these three methods only were submitted this year, in the hope that the results obtained would be such as to warrant their final adoption as provisional in this meeting.

Of each extract two samples were submitted, one of full standard strength, as directed in U. S. Bureau of Chemistry Circular 19, and the other one-fourth that strength. The nutmeg extracts, therefore, contained 2.0% and 0.5% of oil, while all the others contained 3.0% and 0.75%. All were made with 95% alcohol, which gives the test of the methods the maximum of severity. In the case of the weaker extracts this was perhaps more severe than necessary, since the lower grades of extracts are usually made with more or less dilute alcohol.

SAPONIFICATION METHOD FOR WINTERGREEN EXTRACT.

In connection with the saponification method for wintergreen extract, Mr. C. B. Gnadinger suggested titrating instead of weighing the separated salicylic acid. Details for this operation were added to the method as previously studied, and collaborators were requested to express an opinion as to their preference. It seems that the same idea had occurred to other operators, and that essentially the same details have already been in use in other laboratories. The results reported and the comments submitted follow:

Analyses of wintergreen extract.

COLLABORATORS	OIL PRES- ENT	OIL FOUND	
		Gravimetric	Volumetric
	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>
C. O. Dodge.....	0.75 3.00	0.78 3.01	0.73 2.90
C. B. Gnadinger.....	0.75 3.00	0.76 0.76 2.99 2.98	0.75 0.75 2.94 2.94
C. F. Jablonski.....	0.75 3.00	0.74 0.74 2.96 2.97	0.73 0.73 2.93 2.94
H. L. Lourie.....	0.75 3.00	0.79 0.77 2.98 2.98
J. P. Street.....	0.75 3.00	0.80 0.74 3.01 2.97
C. F. Sutton.....	0.75 3.00	0.76 0.74 2.99 2.99	0.76 0.74 2.98 2.98
A. R. Todd.....	0.75 3.00	0.90 3.06	0.80 2.97
P. B. Yost.....	0.75 3.00	0.77 0.80 3.02 3.05	0.78 3.06
Averages.....	0.75 3.00	0.77 3.00	0.76 2.96

COMMENTS BY COLLABORATORS.

C. B. Gnadinger: Method very satisfactory. Little choice between gravimetric and volumetric modifications. I prefer the latter because it is slightly shorter.

Chas. F. Jablonski: Suggest combining the two methods by titrating the weighed residue.

C. E. Morrison (J. P. Street): The salicylic acid was determined gravimetrically, which method is preferred in this laboratory.

Clarence F. Sutton: Titrated the weighed salicylic acid.

A. R. Todd: The method appears to be very satisfactory. It seems to me that the amount of water used in transferring the solution to a separatory funnel could be cut down to 15 cc.

P. B. Yost: I prefer the original method of weighing, as I find end point in the titration to be rather indefinite.

Analyses of anise and nutmeg extracts.

COLLABORATORS	ANISE			NUTMEG		
	Oil present		Oil found	Oil present		Oil found
	per cent	per cent		per cent	per cent	
E. H. Berry.....	0.75	0.8	0.8	0.5	0.5	0.5
	3.00	3.2	3.2	2.0	2.0	2.0
C. L. Black.....	0.75	0.75	0.8	0.5	0.5	0.6
	3.00	3.1	3.1	2.0	2.0	2.0
E. Bloomberg.....	0.75	(¹)	(¹)	0.5	(¹)	(¹)
	3.00	3.4	3.4	2.0	2.0	2.0
Jacob Feldbaum.....	0.75	0.8	0.8	0.5	0.6	0.5
	3.00	3.0	3.2	2.0	1.8	2.0
C. B. Gnadinger.....	0.75	0.7	0.7	0.5	0.5	0.4
	3.00	3.0	2.9	2.0	1.9	1.9
E. J. Munch.....	0.75	1.0		0.5	0.8	
	3.00	3.2		2.0	2.2	
L. Patton.....	0.75	(¹)	(¹)	0.5	(¹)	(¹)
	3.00	3.4	3.4	2.0	2.0	2.0
E. W. Thornton.....	0.75	0.9		0.5	0.2	
	3.00	3.2		2.0	2.0	
G. W. Trainor.....	0.75	1.0	1.0	0.5	0.8	0.8
	3.00	3.2	3.0	2.0	2.2	2.1
Averages.....	0.75	0.83		0.5	0.56	
	3.00	3.2		2.0	2.01	

¹ See comments.

COMMENTS BY COLLABORATORS.

C. L. Black: I found it necessary to use C. P. NaCl, and that even nicer oil layers are obtained by using half saturated sodium sulphate solution.

L. Patton and E. Bloomberg: Stated that the oily layer formed in the case of the dilute extracts did not cover the brine, and that, therefore, an exact reading was not possible; that, however, the amount of oil present is more than a mere trace.

C. B. Gnadinger: Without being advised as to the amount of oil actually present, the results appear to be excellent. If they are reasonably correct, I believe the method should be made official.

E. W. Thornton: The separation in each case was satisfactory without centrifuging a third time.

Analyses of spearmint, peppermint, and wintergreen extracts.

COLLABORATORS	OIL PRES- ENT	OIL FOUND			
		Spearmint	Peppermint	Wintergreen	
E. H. Berry.....	0.75	0.8	0.8	0.8	
	3.00	3.0	3.0	3.1	
R. D. Cook.....	0.75	0.9	0.9	0.8	
	3.00	3.0	3.1	2.7	
C. B. Gnadinger.....	0.75	0.7 0.8	0.8 0.8	0.8 0.8	
	3.00	2.8 2.9	3.0 2.9	3.0 3.0	
J. S. McCune.....	0.75	0.7 0.8	0.8 0.8	0.7 0.7	
	3.00	3.0 3.0	3.0 3.0	3.0 2.8	
E. J. Munch.....	0.75	0.8 0.8	0.6 0.6	0.8 0.8	
	3.00	2.8 2.9	3.0 3.0	3.0 3.0	
K. J. Osterhout.....	0.75	0.7 0.8	0.8 0.8	0.8 0.8	
	3.00	2.8 2.7	3.0 3.0	3.0 3.0	
G. W. Trainor.....	0.75	0.9 1.0	0.9 0.9	0.9 0.9	
	3.00	2.8 3.0	3.0 3.0	2.9 2.9	
Averages.....	0.75	0.8	0.8	0.8	
	3.00	2.9	3.0	2.9	

COMMENTS BY COLLABORATORS.

E. H. Berry: This method worked very satisfactorily, as it did last year. It certainly should be made official.

C. B. Gnadinger: I think the method is an excellent one, and should be made official.

K. J. Osterhout: Thinks that there may be some volatilization of oil in the case of spearmint.

CONCLUSIONS.

The results throughout are quite satisfactory. The saponification method for wintergreen yielded results which are truly remarkable in accuracy and in concordance. As to the details for determining the amount of salicylic acid separated, there seems to be very little choice between the gravimetric and volumetric procedure. The gravimetric results appear to be a little closer, and it would seem preferable to retain the method in its original form.

In the brine method for anise and nutmegs, the results for the standard extracts are excellent. Even for the dilute preparations, the reports are very satisfactory.

The carbon bisulphid method for peppermint, spearmint, and wintergreen yielded most satisfactory results.

Referring particularly to wintergreen extract, it would seem desirable to adopt both the saponification method and the bisulphid method, for the reason that the former gives the percentage of actual methyl salicylate present, while the latter gives the percentage of oil. The presence of a foreign oil, therefore, will become apparent if both methods are used. It is suggested, however, that the saponification method be described as a method for methyl salicylate.

RECOMMENDATIONS.

In view of the above results, supplementing and corroborating the outcome of last year's study on flavoring extracts, the following recommendations are respectfully submitted:

(1) That the saponification method of Hortvet and West for methyl salicylate in wintergreen extract, as described in the Journal of Industrial and Engineering Chemistry, 1909, No. 1, and slightly modified in U. S. Bureau of Chemistry Bulletin 152, page 141, by the then associate referee, R. S. Hiltner, be adopted as provisional. The method follows:

Mix 10 cc. extract in a 100 cc. beaker with 10 cc. of potassium hydroxid solution (10%). Heat on a boiling water bath until volume is reduced about one-half. Add a distinct excess of dilute hydrochloric acid, cool, and extract with three portions of ether, 40 cc., 30 cc., and 20 cc., respectively. Filter the combined ether extracts through a dry filter into a weighed dish, wash with 10 cc. ether, and evaporate spontaneously. Dry over calcium chloride in a desiccator and weigh. The weight of salicylic acid thus obtained multiplied by 9.33 gives the percentage of oil of wintergreen by volume.

(2) That the following method, devised by Hortvet and West, and described in the Journal of Industrial and Engineering Chemistry, volume 1, No. 1, be made provisional for anise and nutmeg extracts:

To 10 cc. extract in a Babcock milk flask add 1 cc. of hydrochloric acid (1 : 1), then sufficient half-saturated salt solution previously heated to 60°C. to fill the flask nearly to the neck. Cork and let stand in water at 60°C. for about 15 minutes, occasionally giving the flask a twisting motion, and centrifuge for 10 minutes at about 800 revolutions per minute. Add brine till the oil rises into the neck of the bottle, and again centrifuge for ten minutes. If the separation is not satisfactory, or the liquid is not clear, cool to about 10°C. and centrifuge for an additional ten minutes. Multiply the reading by 2 to obtain the percentage of oil by volume.

(3) That the following slight modification of the Howard-Mitchell method, which has been studied during the last two years, be now provisionally adopted for peppermint and spearmint extracts and for the determination of oil in wintergreen extract. The method follows:

Pipette 10 cc. of the extract into a Babcock milk bottle, add 1 cc. of carbon disulphid, mix thoroughly, then add 25 cc. of cold water and 1 cc. concentrated hydrochloric acid. Close the mouth of the bottle with the thumb and shake vigorously, whirl the bottle in a centrifuge for six minutes, and remove all but 3 or 4 cc. of the supernatant liquid, which should be practically clear, by means of a glass tube of small bore, and aspiration.

Connect the stem of the bottle with a filter pump, immerse the bottle in water kept at approximately 70°C. for three minutes, removing from the bath every 15 seconds and shaking vigorously. Continue in the same manner for 45 seconds, using a boiling water bath. Remove from the bath and shake while cooling.

Disconnect from the suction and fill the bottle to the neck with saturated salt solution at room temperature, centrifuge for two minutes, and read the volume of the separated oil from the top of the meniscus. Multiply the reading by 2 to obtain the percentage of oil by volume.

In the case of wintergreen, use as floating medium a mixture of 1 volume of concentrated sulphuric acid and 3 volumes of saturated sodium solution.

Since submitting the above report on flavoring extracts, two further collaborators submitted their reports. The first was by A. G. Woodman, of Boston, whose results by the methods submitted are similar to those obtained by the other collaborators, but he makes a very interesting statement regarding a nephelometric method which he has tried on very dilute extracts, and he reported his results on the dilute samples of anise and nutmeg submitted. His results were:

	OIL PRESENT	OIL FOUND
	<i>per cent</i>	<i>per cent</i>
Anise.....	0.75	1.00
Nutmeg.....	0.50	0.50

While Mr. Woodman's details were not available, a few simple tests were made along the line suggested, and it is believed that the idea is promising and that when Mr. Woodman's paper is available it may be desirable to study the method for use on very dilute preparations.

The second paper received was by C. O. Dodge, of the Bureau of Chemistry in Washington. His results on the wintergreen extracts by the saponification method, slightly modified by himself, are highly accurate and are as follows:

	OIL PRESENT	OIL FOUND
	<i>per cent</i>	<i>per cent</i>
Wintergreen extracts:		
No. 1	0.75	0.75
No. 2	3.00	2.99

Mr. Dodge suggests that the details submitted may be improved by keeping down the bulk of the solution to be extracted and using a smaller amount of ether. However, the details suggested by him involve four extractions with a total of 60 cc. of the solvent, while the original method requires only three extractions with a total of 90 cc. of ether. Inasmuch as the results obtained by the collaborators were very satisfactory, there is doubt that the changes are necessary.

Mr. Dodge raises the further point that oil of wintergreen may contain only 98% methyl salicylate, and suggests that the calculation of the result should be based on this figure. It seems, however, that this objection is overcome by the recommendation that this determination be considered a determination of methyl salicylate rather than a determination of oil.

REPORT ON BAKING POWDERS.

By H. E. PATTEN (Bureau of Chemistry, Washington, D. C.),
Associate Referee.

Tests of three gravimetric methods for the determination of lead in phosphate baking powders were made. Five analysts coöperated. The methods studied were the Seeker-Clayton, Remington modification of the Seeker-Clayton, and Exner methods. Directions for these methods were sent out as follows:

DESCRIPTION OF METHODS.

SEEKER-CLAYTON METHOD.

Place 20 grams of the baking powder in a large beaker and add about 25 cc. of water in small portions at a time to avoid excessive frothing. Add 20 cc. of concentrated hydrochloric acid, a little at a time for the same reason, and digest on a steam bath until the solution is perfectly clear and limpid or until a drop of the solution gives no reaction for starch with iodine and potassium iodide solution. Add sufficient solution of ammonium citrate¹ (lead-free) to correspond to 20 grams of citric acid and render slightly alkaline to litmus with ammonia, density 0.95. The latter is added a little at a time, with care to keep the solution cool, to avoid precipitation of calcium salts. Dilute to about 400–450 cc., add 10 cc. of 10% hydrochloric acid, cool to room temperature, saturate with hydrogen sulphid, and allow to stand overnight.

Filter through a moistened filter, using suction if necessary, and wash the precipitate² with hydrogen sulphid water and finally with a little water. Dissolve the precipitate by passing through the filter three 5 cc. portions of boiling 10% hydrochloric acid followed by three 5 cc. portions of boiling 25% nitric acid, and collect the filtrate in a 100–150 cc. beaker. Finally wash the filter with a little hot water, add 2 cc. of concentrated sulphuric acid to the filtrate and washings, and evaporate on a hot plate until fumes of sulphuric acid are copiously evolved. The solution should now be practically colorless, but if not so add a little nitric acid and again evaporate until fumes appear. Cool, add 10 cc. of water and 20 cc. of alcohol, and allow to stand overnight. Filter through a Gooch on asbestos and wash with alcohol.

¹ *Ammonium citrate solution.*—This reagent may be prepared by dissolving 100 grams of citric acid in 100 cc. of hot water, cooling, adding a little at a time sufficient ammonia to leave a slight excess, again cooling, and then saturating with hydrogen sulphid. Allow to stand overnight, or until the sulphids have settled out, filter, boil the filtrate to expel excess of hydrogen sulphid and ammonia, cool, and make up to 200 cc. Lead-free citric acid may be used instead of this solution, but it has the disadvantage of causing a considerable evolution of heat in the subsequent neutralization with ammonia, resulting in a precipitate of calcium citrate.

² In some cases, especially when a very large amount of calcium phosphate is present, a white precipitate of calcium citrate will settle out on standing overnight. In such a case, decant the supernatant liquid through a filter and dissolve the precipitate in a small amount of dilute hydrochloric acid, add an excess of ammonium citrate, cool, render slightly alkaline with ammonia, cool, saturate with hydrogen sulphid, and allow to stand from 6 to 12 hours. The precipitated sulphids are then filtered off and treated as in the regular method. (The material remaining on the filter from the liquid first decanted has in the meantime been washed with hydrogen sulphid water and dissolved in hot hydrochloric and nitric acid, the solution being finally combined with that from the second crop of lead sulphid.)

Place the Gooch in a small beaker and treat the contents with a few drops of concentrated ammonia. Then pour 10 cc. of 50% ammonium acetate into the crucible and allow it to stand for about 15 minutes. Remove the crucible from the beaker and carefully wash the bottom and sides with water, allowing the washings to run into the beaker. Now, by placing the lips over the top of the crucible, blow the solution still remaining in the crucible into the beaker. Wash the crucible with a little water, forcing the washings through the asbestos pad in the manner just described. Rinse the bottom of the crucible with a jet of water and fit it into a bell jar arranged for filtering by suction. Filter the contents of the beaker through the Gooch, collecting the filtrate in a second beaker placed under the bell jar, and wash thoroughly with hot water. Acidify the filtrate with acetic acid, heat nearly to boiling, add an excess of potassium bichromate, and allow to stand overnight. Then filter through a tared Gooch, dry for 20-30 minutes on a hot plate, cool, and weigh as PbCrO_4 .

REMINGTON MODIFICATION OF THE SEEKER-CLAYTON METHOD.

Weigh 100 grams of the baking powder into a large beaker (1.5-2 liters) and add 500 cc. of the cold citrate solution¹ slowly with stirring. If the powder contains albumin a troublesome frothing may result, which can be controlled by the use of a few cubic centimeters of ether. Stir thoroughly several times, and set aside for several hours.

Decant the supernatant liquid, and wash the starch twice by decantation with about 50 cc. of water, adding the washings to the original solution. (A centrifuge will be of great assistance in the decantation and washing.) Heat the solution on the steam bath until the albumin is coagulated, and pass in a slow stream of hydrogen sulphid for several hours, or overnight. (If a heavy white precipitate is formed, add ammonia cautiously until it is dissolved, taking care that the solution remains acid.) Filter off the precipitated sulphids and wash with water containing hydrogen sulphid.

To the residue of starch add 250 cc. of water and 25 cc. of hydrochloric acid, and heat on the steam bath until the starch is completely hydrolyzed. Neutralize with ammonia, and make just acid to litmus with hydrochloric acid. Pass in a slow stream of hydrogen sulphid for several hours, or overnight. Filter and wash with water containing hydrogen sulphid.

Place the filters containing both precipitates in a tall 100 cc. beaker, add 5 cc. of sulphuric acid and 10 cc. of nitric acid, and heat on the hot plate, with frequent additions of nitric acid until all organic matter is destroyed and the liquid is nearly colorless. Take down to fumes of sulphuric acid, cool, add 20 cc. of water and 40 cc. of alcohol, and allow to stand overnight.

From this point proceed as in the original Seeker-Clayton method, as follows:

Filter through a Gooch on asbestos, and wash with alcohol. Place the Gooch in the original beaker and moisten the contents with a few drops of concentrated ammonia. Then pour 10 cc. of 50% ammonium acetate solution (previously treated with H_2S and boiled) into the crucible and allow it to stand for about fifteen minutes. Remove the crucible from the beaker and carefully wash the bottom and sides with water, allowing the washings to run into the beaker. Now, by placing the lips over the top of the crucible, blow the solution still remaining in the crucible

¹ *Preparation of ammonium citrate reagent.*—Dissolve 200 grams of ammonium citrate in a liter of water, add 10 cc. of concentrated hydrochloric acid, warm slightly, and pass in a slow stream of hydrogen sulphid for several hours. Filter and heat to boiling to expel excess of H_2S .

into the beaker. Wash the crucible with a little water, forcing the washings through the asbestos pad in the manner just described. Rinse the bottom of the crucible with a jet of water, and fit it into a bell glass fitted for filtering with suction. Pass the ammonium acetate solution through the Gooch, filtering twice if necessary to secure a perfectly clear filtrate, and wash thoroughly with a little hot water. Acidify the filtrate with acetic acid, heat nearly to boiling, add an excess of potassium dichromate, and allow to stand overnight. Filter on a small tared Gooch, wash the precipitated lead chromate with cold water, dry the crucible and contents by heating for 20-30 minutes on a hot plate, cool, and weigh as PbCrO_4 .

EXNER METHOD.

Weigh out 200 grams¹ of the sample and transfer this to a 3-liter Jena flask. Add 300 cc. concentrated nitric acid in portions, with thorough shaking after each addition. The mixture, which first forms a thick paste, is slowly heated on an asbestos gauze with repeated shaking. The mass becomes thinner, and by the time the oxidation of the starch begins it is quite fluid. As soon as brown fumes begin to appear at the mouth of the flask, heat is removed and a stemless funnel is inserted in the neck of the flask. The reaction soon becomes very vigorous, and the flask should be set in a hood with a good draft. The reaction, however, will not become so violent as to cause loss of material by foaming over. When the action has moderated, the flask is set back on the asbestos gauze over a moderate Bunsen flame. When the action becomes weak, as shown by the fumes in the flask becoming lighter, 90 cc. concentrated sulphuric acid is slowly added to the contents of the flask, and heating is again continued until the fumes fade. Then 25 cc. of concentrated nitric acid are added from time to time, with continued heating, until all the starch is completely oxidized. This can be told from the behavior of the last addition of nitric acid, which boils out with but little decomposition. Usually three or four additions of 25 cc. portions of nitric acid suffice. The nitric acid should finally be expelled as completely as possible without endangering the flask. Cool and add 400 cc. distilled water, shake, and allow to settle. The soluble sulphates of sodium, potassium, aluminum, iron, etc., go into solution, while calcium sulphate and most of the lead sulphate will be precipitated. Filter through an 18 cm. folded filter into a liter Erlenmeyer flask; rinse the 3-liter flask two or three times with small portions of water and pour the rinsings through the filter. There is no need of a thorough washing of the precipitate. The latter is transferred to a 2-liter Erlenmeyer flask. This is best done by opening the filter containing the precipitate over a 600 cc. beaker and rinsing thoroughly with water from a wash bottle. The contents of the beaker are then transferred to the Erlenmeyer, together with whatever precipitate remains in the 3-liter flask. The contents are then diluted so as to nearly fill the flask, stirred thoroughly to dissolve the calcium sulphate, 20 cc. strong acetic acid added, and the liquid thoroughly saturated with hydrogen sulphid. This may be done quickly by preparing a solution of sodium sulphid by saturating a 5% sodium hydroxid solution completely with hydrogen sulphid, drawing up this liquid into a pipette, which is then dipped into the solution in the Erlenmeyer and stirred while the sulphid runs out. Only a small part of the free acid need be neutralized to produce complete saturation with hydrogen sulphid. This procedure saves both time and gas. The flask is then corked and set aside until the precipitate has settled. The liquid is then siphoned off, saving much time in filtering. The lower end of the siphon tube which is introduced into the liquid has a short bend at right angles so

¹ If other weights have to be used, take reagents in proportion.

that it will not suck the precipitate from the bottom. When there is much calcium sulphate, one such treatment will not suffice to bring all into solution, but the flask is refilled with distilled water, is again acidified and saturated with hydrogen sulphid and allowed to settle till the calcium sulphate is practically all dissolved and the residue of sulphids is dark colored. When much calcium sulphate is present, solution may be hastened by the addition of lead-free sodium acetate to the water, 50-75 grams to each 2 liters of water. If the solution of the salt gives no darkening with hydrogen sulphid, it may be safely used.

The liquid containing the soluble sulphates is treated separately to recover the trace of lead which it may contain. The acidity is partially neutralized with ammonium hydroxid just short of the point of producing a permanent precipitate of aluminum phosphate. It is then saturated with hydrogen sulphid and the precipitate allowed to settle. Some iron sulphid will usually be precipitated also. It is important that the sulphid precipitations be made in very slightly acid solutions, otherwise lead sulphid will not be completely precipitated. When both precipitates have finally settled, the liquids may be siphoned off, and the precipitates transferred on to separate 11 cm. filters and washed with hydrogen sulphid water. The filters with their precipitates are placed into a 200 cc. Erlenmeyer flask, 10 cc. concentrated nitric acid and 5 cc. concentrated sulphuric acid are added, a stemless funnel inserted in the neck, and the flask heated to completely oxidize the material. When the nitric acid has all been expelled and the residue darkens, more nitric acid is added until no such darkening occurs. The residue is finally heated till fumes of sulphur trioxid are given off. Then cool and add 15 cc. of water. Filter through 7 cm. filter, rinse, and then wash the filter twice with small portions of dilute sulphuric acid, and finally with a little water. There is no need of removing all sulphuric acid. Place a clean 150 cc. beaker under the filter, dissolve the precipitate with 15-25 cc. ammonium acetate and wash thoroughly with water. The ammonium acetate is best prepared by mixing 1 part 99% acetic acid, 1 part water, and 1 part ammonium hydroxid sp. gr. 0.90, introducing a piece of litmus paper, and cautiously adding more ammonium hydroxid until neutral.

The solution of lead in ammonium acetate is acidified with acetic acid and precipitated with potassium bichromate. The mixture is heated on the steam bath and then allowed to cool and settle. It is filtered on to a tared Gooch having a thick felt, washed with water, dried at about 125°C., and weighed as lead chromate.

It is taken as a matter of course that the water used and the reagents must be free from lead. There seems to be no danger from lead in vessels when Jena glass is used.

SAMPLE SOLUTIONS.

In view of the lack of confidence expressed by many analysts with whom we consulted in the Seeker-Clayton method as originally given, it was decided to send out for a preliminary test samples containing the lead in a soluble form. A very slight amount of organic material was added to necessitate wet oxidation as recommended by the methods. Some of the collaborators very properly objected on the ground that these samples did not offer the same analytical difficulties met with in the analysis of phosphate baking powders. They have, nevertheless, worked faithfully upon the samples and have sent in data which, in the judgment of the associate referee, fully justify the style of sample used

for this preliminary test of the comparative merits of the methods. Further, to eliminate personal equations and any autosuggestion in the analysts, three sample solutions labeled A, B, and C were sent out, each containing exactly the same concentration of lead (as $\text{Pb}(\text{NO}_3)_2$), but without indicating to the analysts that these solutions were duplicates one of the other so far as the lead was concerned. The ingredients of the solutions are given below.

It will be noted that the diluted solutions made from these samples which were finally to be taken for the lead tests contained a quantity of lead the same as would be found in a baking powder containing 20 parts of lead per million. Thus, 200 grams of baking powder (Exner method) containing 20 parts per million of lead would have 4 mg. of lead. One hundred grams (Remington method) would contain 2 mg. of lead, and 20 grams (Seeker-Clayton method) would contain 0.4 mg. The solutions measured out for analysis contained these respective amounts.

INGREDIENTS OF SOLUTIONS.

Solution A contained 10 grams of sugar per 100 cc. and 40 mg. of lead per 100 cc. (as $\text{Pb}(\text{NO}_3)_2$), with a few drops of concentrated nitric acid to repress hydrolysis.

Solution B was an exact duplicate of solution A and was taken from the same mix.

Solution C was identical with solutions A and B except that 2.5 grams $\text{CaH}_4(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$ (found to be lead free) was added per 100 cc.

DIRECTIONS ACCOMPANYING SAMPLE SOLUTIONS.

The following directions were sent out with the solutions:

Take 50 cc. of each solution at 25°C. and make up with distilled water to 500 cc. at 25°C. Then for use in the Exner method use 100 cc. of this diluted solution; for the Remington method use 50 cc.; and for the Seeker-Clayton method use 10 cc. The results should be reported in milligrams per liter of original solution.

REPORTS OF COLLABORATORS.

The collaborators reported results which are tabulated as milligrams per liter of original sample solution in Table 1. These results have been recalculated to a basis of parts per million of a phosphate baking powder, using the weights of baking powder recommended for analysis in each of the methods, respectively, and are given in Table 2.

One of the collaborators, Dr. T. J. Bryan, in addition to the requested work, added to the diluted solutions to be taken for analysis an amount of lead-free baking powder required by the different methods and then

TABLE 1.

Lead in milligrams per liter of original solution as reported by the collaborators.
 [Solutions contained 400 mg. of lead per liter.]

COLLABORATOR AND ANALYST	LEAD IN MILLIGRAMS PER LITER OF SOLUTION								
	Solution A			Solution B			Solution C		
	Seeker-Clayton	Remington	Exner	Seeker-Clayton	Remington	Exner	Seeker-Clayton	Remington	Exner
Loomis (Wichman)...	128	333	333	128	307	358	128	422	416
Hortvet (Pettijohn)...	1220	640	870	640	500	370	380	460	420
	2750	990	700	380	450	210
			700						
Morey (Hoyt).....	260	550	359	380	360	436	260	396	378
	380	475	372	320	422	429	190	410	359
Bryan.....	384	371	454	833	358	211	1020	269	358
	897	294	371	512	179	237	1020	294	397
	513	436	442	513	410	410	448	576	481
Stallings (Clarke)....	448	422	416	448	422	384	513	512	512
	385	448	577
				422					

TABLE 2.

Recalculation of results given in Table 1 to a basis¹ of parts per million in a phosphate baking powder.

[Solutions contained 20 parts of lead per million.]

COLLABORATOR AND ANALYST	LEAD IN PARTS PER MILLION								
	Solution A			Solution B			Solution C		
	Seeker-Clayton	Remington	Exner	Seeker-Clayton	Remington	Exner	Seeker-Clayton	Remington	Exner
Loomis (Wichman)...	6	17	17	6	15	18	6	21	21
	61	32	44	32	25	19	19	23	21
Hortvet (Pettijohn)...	138	50	35	19	22	11
				35					
Morey (Hoyt).....	13	28	18	19	18	22	13	20	19
	19	24	19	16	21	21	10	21	18
Bryan.....	19	19	23	42	18	11	51	13	18
	45	15	19	26	9	12	51	15	19
	26	22	22	26	21	21	22	29	24
Stallings (Clarke)....	22	21	21	22	21	19	26	26	26
	19	22	22	29
				21					

¹ For synthesis of samples see page 218.

TABLE 3.

Lead found in solutions to which lead-free baking powder was added in the proportions required by the various methods.

[Solutions contained 400 mg. of lead per liter.]

METHOD	LEAD IN MILLIGRAMS PER LITER OF SOLUTION		
	Solution A	Solution B	Solution C
Seeker-Clayton	704	320	384
	448	192	192
Remington.....	410	179	102
	397	38	140
Exner.....	134	121	313
	281	294	

subjected the solutions to analysis. These results are shown in Table 3. Table 4 contains the results given in Table 3 recalculated to parts per million of lead in a phosphate baking powder in order to make them comparable with similar results given in Table 2.

TABLE 4.

Recalculation of results of Table 3 to a basis of parts per million in the phosphate baking powder added.

[Solutions contained 20 parts of lead per million.]

METHOD	LEAD IN PARTS PER MILLION		
	Solution A	Solution B	Solution C
Seeker-Clayton.....	35	16	19
	22	10	10
Remington.....	22	9	5
	20	2	7
Exner.....	7	6	16
	14	15	

DISCUSSION OF RESULTS.

Recurring now to the objections which have been made by some of the collaborators (H. M. Loomis, T. J. Bryan, C. B. Morey): They were of the opinion that these methods for the estimation of lead should be used upon lead-free baking-powder samples to which known quantities of lead had been added, or upon sample solutions containing the same quantity and ratio of ingredients that would occur upon solution of a phosphate baking powder in acid.

These methods have two objects: One, to determine the lead quantitatively, and the other to separate the lead from the mixture in which it is found. It seemed advisable to study first their respective merits in determining lead under conditions where there was practically no interference from baking-powder ingredients. If a method can not stand this test, manifestly there is no use of trying it out where there is added the further difficulty of separating the lead from a complex mixture of ingredients.

The reason that the associate referee sent out these very simple samples for analysis was to ascertain if these three methods could give comparable results in the hands of capable analysts under the most simple conditions. If this had proved to be the case, it was then the intention to carry out further work upon more complicated samples fully simulating the actual conditions found in phosphate baking powders. Owing to the lateness of the date at which the present associate referee took up this work, it has been possible to proceed only as far as the report indicates.

In the work done by Mr. Pettijohn, under Dr. Hortvet, the prescribed

methods were not followed exactly in all cases; and for this reason, together with the wide variation in results presented, it seems fair to judge the methods on the results reported by the other four analysts.

SEEKER-CLAYTON METHOD.

VARIATIONS REPORTED.

The Seeker-Clayton method might possibly be adapted for this work by substituting a colorimetric reading at the end instead of the present gravimetric determination, but in its present form it gives results varying from (Table 2) 6 parts per million to 51 parts per million, using one solution with four different analysts, and from 19 parts per million to 51 parts per million for one analyst on three solutions (Bryan).

WEIGHING LIMITATION ERROR.

The inherent difficulty with the Seeker-Clayton method is the small amount of sample taken. With the baking-powder standard for lead set at 20 parts per million upper limit, there will be 0.0004 gram of lead in a 20-gram sample containing this highest limit. Since the lead is weighed as PbCrO_4 , the actual mass weighed would be 0.000624 gram. Assuming that the balance weighs accurately 0.0001 gram, then any weight between 0.00055 gram and 0.00065 gram will be read as 0.0006 gram of PbCrO_4 . This weight calculated to lead gives 0.000385 gram lead, with a range from 0.000353 gram to 0.000417. Calculating to parts per million of lead, 0.0006 gram PbCrO_4 represents 19.25 parts per million, and the range is 3.2 parts per million (from 17.65 to 20.85). Hence we have a possible error of $\pm 8.3\%$ inherent in the limitations of the balance when estimating lead in baking powders containing approximately 20 parts per million, even when the balance weighs accurately to 0.0001 gram. The above standard referred to (20 parts per million of lead) is for baking-powder acid-ingredient chemicals. The standard for complete baking powders is one-half of this, or 10 parts per million. A baking powder containing this upper limit would yield only 0.0003 gram PbCrO_4 and, the weighing errors remaining the same, the percentage of error due to the limitations of the balance would be doubled.

WEIGHT STANDARDIZATION ERROR.

The above discussion of weighing errors assumes that the set of metric weights used with the balance is rigidly standardized, and that each correction of standardization is used throughout all operations. To illustrate how great such an error may be, test 11388, U. S. Bureau of Standards, of a new set of weights (Becker's Sons, Rotterdam) showed the following: One of the 2-gram weights was too heavy by 0.1 mg., each 100-mg. weight was 0.04 mg. too light, and the 50-mg. weight was 0.02

mg. too light. Thus, a weighing in which one of the 2-gram weights was replaced by the other would show 0.0001 gram more or less than the correct mass. Similarly, a weighing in which the two 100-mg. weights and the 50-mg. weights were used together would lack 0.0001 gram of the true mass. Thus the possible error through neglecting the standardization of weights would be as large as the above mentioned error due to the limitations of the balance. And even when weight standardization is taken care of, constant use and corrosion of weights may introduce error.

OPTIMUM LOAD.

In making such close estimates as required by the Seeker-Clayton method, the effect of the balance load on the sensibility must be taken into account. It would, of course, be advisable to determine the load of greatest sensibility, and then add weights to the pan containing the Gooch crucible until this optimum load is approximated; or a lighter crucible might be chosen in case the optimum load were lower.

DRYING ERROR.

The method of drying the PbCrO_4 precipitate on the hot plate according to the Seeker-Clayton directions has been found to be subject to an error of 0.0002 gram by Dr. T. J. Bryan in his report, although he does not specify exact reasons for this error. Mr. Pettijohn also has criticized this procedure, but without quantitative expression. This error may be partially due to absorption of moisture during the weighing or in the balance case.

RECOMMENDATIONS ON SEEKER-CLAYTON METHOD.

The Seeker-Clayton method, because of the small quantity of sample and reagents required, may well serve as a semiquantitative method in the hands of one skilled in its use; but taking into account all of the factors mentioned, it would seem inadvisable to recommend the Seeker-Clayton method for further study.

REMINGTON MODIFICATION.

VARIATIONS REPORTED.

The Remington modification method showed much closer checking than the Seeker-Clayton, the greatest variation being from 13 to 29 parts per million with one solution and four different analysts, and from 9 to 19 parts per million with three solutions for one analyst (Bryan).

The majority of determinations, however, came reasonably close to the true value of 20 parts per million. Out of a total of 23 determinations by four analysts, over half (13) were within 10% of the correct value. Fifteen were within 20%, and four showed an error greater than 30%.

WEIGHING ERRORS.

With this method the larger weight of sample taken (100 grams) reduces the error in weighing due to the limitations of the balance to ± 0.32 parts per million when a total of 20 parts per million is being estimated. Other weighing errors are correspondingly reduced. An error of 0.0001 gram in weighing the PbCrO_4 would produce an error of 0.64 parts per million with the 20 parts per million total.

RECOMMENDATIONS ON REMINGTON MODIFICATION.

From the results obtained with this method, it would appear that the Remington method does not possess quite the accuracy of the Exner method. Consequently it is recommended that no further study be made of the Remington modification method at present.

EXNER METHOD.

VARIATIONS REPORTED.

Still closer checking was found in the results with the Exner method. The greatest variation was from 11 to 22 parts per million with one solution and four analysts, and from 11 to 23 parts per million with one analyst (Bryan), using three solutions. Fifteen out of a total of 21 determinations were within 10% of 20 parts per million, and only three showed an error greater than 20%.

WEIGHING ERRORS.

With the Exner method weighing errors are still further reduced, the error due to weighing limitations of the balance being only ± 0.16 parts per million in estimating 20 parts per million of lead. An error of 0.0001 gram in weighing the lead chromate precipitate would give an error of 0.32 parts per million for this 20 parts per million total.

USE OF ALCOHOL IN PRECIPITATING LEAD SULPHATE.

Mr. Pettijohn recommends that alcohol be added in the Exner method, at the point where the PbSO_4 (recently formed from the sulphid by the action of nitric and sulphuric acids) is treated with water, in order to precipitate the lead sulphate completely. It appears to the associate referee that this would delay the determination and not add appreciably to the accuracy. The solubility of PbSO_4 in water is 0.0042 gram PbSO_4 per 100 grams distilled water at 19°C . (Rothmund: Löslichkeit und Löslichkeitsbeeinflussung). This means that when *saturated*, 15 cc. of water would hold only 0.0006 gram of PbSO_4 . The 15 cc. of water used in transferring the sulphate precipitate does not have time to reach saturation; hence the use of alcohol to recover this minute quantity of PbSO_4 .

is without material effect upon the accuracy of the method in general, if carried out according to Exner's directions. However, where it is desired to use the Exner method with fractional quantities of the 200-gram sample, the error introduced by the loss of the PbSO_4 in the transference water might need consideration.

RECOMMENDATIONS ON THE EXNER METHOD.

In view of the shorter time required and the greater accuracy both from a priori considerations and actual experimental data, the Exner method seems well worthy of further study, where a gravimetric method is to be used. In case the full quantity recommended (200 grams) is used, it is advisable to take the substance from the can, thoroughly mix, and secure each complete sample taken for analysis as a composite of many fractions lifted from different portions of the mix.

WICHMANN MODIFICATION OF SEEKER-CLAYTON METHOD.

Mr. H. J. Wichmann in his report presents the following method, which he recommends as dependable:

One hundred grams of baking powder are weighed out into a 1.3-liter beaker and 10% HCl added to excess in small portions. Excessive frothing may be kept down with ether. The mixture of acid and starch is heated until the starch is hydrolyzed and the solution is quite limpid. After cooling, 200 cc. 50% ammonium citrate solution are added. The solution is kept cold while an excess of ammonia is slowly and carefully added. If a precipitate forms, sufficient ammonium citrate is added to dissolve it. Fifteen cubic centimeters of saturated HgCl_2 solution are added and the solution diluted to about 1,200 cc. Hydrogen sulphid is then passed to saturation and the beaker set aside. The precipitate settles rapidly and can readily be filtered. A centrifuge can be used to advantage. There is no need of standing overnight, as the heavy HgS will drag down the PbS . If the solution stands overnight a precipitate of calcium citrate is apt to settle out.

The filter paper with the sulphids is placed in a small casserole, or the sulphids are washed into it from the cylinder, if a centrifuge is used. Ten cubic centimeters of HNO_3 and 2 cc. of concentrated H_2SO_4 are added. The nitric acid is evaporated and the sulphuric acid slowly fumed off. If a gas muffle furnace is available the casserole can be placed on the platform in front of the muffle until the H_2SO_4 has disappeared and then slowly heated inside to a light red heat. The mercury salts will vaporize, and any ferric sulphate present will be broken up to the oxid. After cooling, the lead sulphate is leached out from the residue with an ammoniacal 25% solution of ammonium acetate. Several leachings must be made. The filter paper is then washed with hot water. The filtrate is acidified with acetic acid and the lead precipitated as chromate with potassium dichromate. After standing overnight the lead chromate is collected on a Gooch, dried by heating to 125°C ., cooled, and weighed.

In answer to the objection that the mercury would tend to carry off some of the lead with it during volatilization, Mr. Wichmann assures

us that he has not found this error to interfere with the accuracy of the method. Mr. Clayton states that in his experience such a volatilization of mercury salts gave low results for the lead; but considering the very much smaller quantities of lead present in each test sample when following the Seeker-Clayton method, it seems likely that the large relative error obtained by Mr. Clayton would become insignificant when using 100-gram samples as recommended by Mr. Wichmann.

RECOMMENDATIONS.

The associate referee recommends that further study be carried out upon the Exner and Wichmann methods. The Exner method is recommended for adoption as a provisional method.

Special emphasis should be laid upon speed of operations, as well as upon clean separations, close measurement of wash water, and accurate weighing. The use of a stirrer during hydrogen sulphid precipitation, followed by centrifuging, should be studied with a view to eliminating the standing overnight. The Wichmann method presents the only gravimetric determination which can be carried out within approximately 24 hours, and is therefore especially commended for careful study. It is recommended that the determinations be accompanied by a time schedule, showing actual time involved in each operation. This is especially important in view of the large number of samples and the short time available in handling many carloads of material at the factories.

The associate referee wishes to thank the collaborators, T. J. Bryan, J. Hortvet, H. M. Loomis, C. B. Morey, and R. E. Stallings, and the analysts, J. O. Clarke, F. L. Hoyt, E. Pettijohn, and H. J. Wichmann, for their helpful spirit in carrying out the work. Acknowledgment is also made to Mr. G. H. Mains for assistance in discussing data and compiling this report.

THE RÔLE OF CALCIUM SULPHATE IN PHOSPHATE BAKING POWDERS.

(ABSTRACT.)

By H. E. PATTEN (Bureau of Chemistry, Washington, D. C.).

The present investigation was undertaken in view of the different opinions which have been expressed as to the function of calcium sulphate in phosphate baking powders. Some chemists hold that the calcium sulphate is merely a neutral inert body acting possibly as a drying agent in the baking powder, but contributing nothing to the chemical changes which alone render the powder of service in baking. Others maintain

that the calcium sulphate acts chemically in the dough, and finally is not found in its original form in the finished biscuit, but broken down chemically and recombined into a soluble product (sodium sulphate) and an insoluble product [calcium phosphate $(\text{CaO})_x(\text{P}_2\text{O}_5)_y$] of variable composition. The presence of calcium sulphate is also held to retard the evolution of gas by the baking powder and thus renders the leavening action more gradual.

There is also question in the minds of some as to whether the reaction between sodium bicarbonate and monocalcium phosphate yields dicalcium phosphate (CaHPO_4) or tricalcium phosphate $\text{Ca}_3(\text{PO}_4)_2$; and also question as to the effect of flour and starch (in the dough of the biscuit) upon the reaction. Indeed some claim that experiments made with sodium bicarbonate, calcium phosphate, calcium sulphate, and water cannot be taken as good evidence of what reactions will take place when these baking-powder ingredients are actually mixed with flour and water and baked into a finished biscuit.

A survey of the literature at the time this work was undertaken¹ showed that accurately controlled heterogeneous equilibria studies had been carried out in which carbonates were investigated as to their action with sulphate and chlorid, but not with phosphate; also that phosphates had been studied in connection with sulphate and chlorid, but not with carbonate. Consequently, there was a large field of reaction possibility still unexplored, even where the baking-powder ingredients alone were taken in aqueous solution, no flour being added.

From these equilibria experiments, and the diagrams graphically representing their data, tentative inferences were made as to the chemical changes which might be expected in the baking reaction, as follows (using symbols for brevity):

If $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ or CaSO_4 anhydrous be added to NaHCO_3 in presence of excess of water—²

(1) CaCO_3 will be formed (since it is about 1,000 times less soluble than CaSO_4) along with Na_2SO_4 and H_2CO_3 .

(2) The H_2CO_3 will then decompose and CO_2 gas will be evolved.

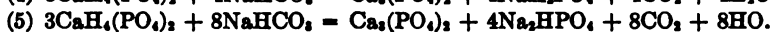
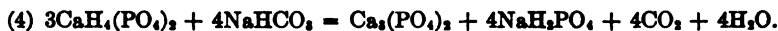
(3) The main effect of rise in temperature above 25°C . is to liberate CO_2 held in solution by CaCO_3 ³, and to deposit the CaCO_3 (released by the CO_2) as a precipitate; also temperature rise changes any residual $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ to the anhydrous form, CaSO_4 .

¹ This problem was referred to the author in 1911, some time in November, by Dr. W. D. Bigelow, at the instance of Dr. H. W. Wiley. A preliminary report was submitted to Dr. Bigelow on January 15, 1912.

² Compare U. S. Patent 316,863, April 28, 1885. CO_2 formed from CaSO_4 + alkaline bicarbonates.

³ $\text{CaH}_2(\text{CO}_3)_2$, calcium bicarbonate has not been shown to exist, despite the frequent references to it in chemical writings.

If $\text{CaH}_4(\text{PO}_4)_2$ be added to NaHCO_3 in water the reaction varies with the quantity of NaHCO_3 used, thus:



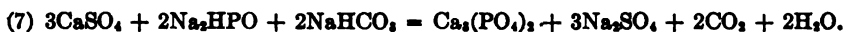
It is found that "tricalcium phosphate" is always formed and the variation of NaHCO_3 produces a variation in NaH_2PO_4 and Na_2HPO_4 and CO_2 and H_2O .

If we add CaSO_4 or $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ to $\text{CaH}_4(\text{PO}_4)_2$ and NaHCO_3 in water, the reaction is modified in that an end-product, Na_2HPO_4 , is acted upon, thus:



The tricalcium phosphate (a solid solution $(\text{CaO})_x(\text{P}_2\text{O}_5)_y$ is about 1,000 times less soluble in water than CaSO_4 , hence this reaction proceeds till the solubility limit of the $\text{Ca}_3(\text{PO}_4)_2$ in the acid solution resulting is reached. This equation, of course, does not represent the reality, since the composition of the solid solution $(\text{Ca}_3(\text{PO}_4)_2?)$ varies with the composition of the liquid phase in contact with it.

Reaction No. 6 above does not take account of the NaHCO_3 present. When this is done we have:



No hint is given in the literature examined of a compound, $\text{CaNa}_4(\text{PO}_4)_2$, either as a solid phase or in solution.

Without regard to exact equations, such as 4, 5, 6, and 7, it may be inferred from the published isotherms referred to in the literature that—

(8) With calcium sulphate present in excess as a solid phase, very little phosphorus pentoxid will be found in solution.

(9) If the content of CaSO_4 is high in the baking powder it is likely that CaSO_4 or $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ will exist as a solid phase after the baking powder reaction is ended, and some CaCO_3 may be present. But if the CaSO_4 content is low in the baking powder, then there would be a solid solution left as residue after baking, containing CaO and P_2O_5 , but not necessarily in a simple molecular ratio to each other; here, too, some CaCO_3 might be left in the solid residue.

(10) Tricalcium phosphate is not permanent or of fixed chemical composition, at least when it is formed in the usual way by precipitation; the P_2O_5 may be washed from it with excess of water, leaving a basic compound, or, conversely, its lime may be extracted by acid solutions, leaving the solid solution relatively higher in P_2O_5 content than in CaO .

(11) The effect of adding salt (sodium chlorid, NaCl) in mixing dough probably is to decrease the P_2O_5 in solution. This effect is observed where chlorids act on calcium phosphates alone in aqueous solution.¹

¹ U. S. Dept. Agr., Bur. Soils Bul. 41, p. 34. KCl decomposes $\text{Ca}_3(\text{PO}_4)_2$ and decreases P_2O_5 in solution.

(12) Precipitation of calcium carbonate in the cold is retarded by the presence of phosphate salts.¹ This would tend, though perhaps only slightly, to hold more CO_2 in solution and give it up at higher temperature in the oven.

The double titration of phosphates, using methyl orange and phenolphthalein and a neutral salt such as calcium chlorid, is outlined in detail by H. N. Morse in his Analytical Chemistry, and has long been a matter of common knowledge. Wadman² in 1894 published results on testing phosphate baking powders, using essentially this method.

Although the foregoing review of the subject makes it appear that calcium sulphate is not inert in the baking-powder reaction, a few experiments have been carried out to ascertain the effect of the calcium sulphate, not only in quantitative experiments with the chemicals and water but also in baking tests.

The following table shows the amount of calcium sulphate not decomposed when varying amounts were used in the baking-powder reaction in flasks with given amounts of monocalcium phosphate and sodium bicarbonate.

Quantity of calcium sulphate left in solid residue after baking-powder reaction.¹

CALCIUM SULPHATE ADDED		CALCIUM SULPHATE LEFT IN SOLID RESIDUE
Grams	Per cent by weight of $\text{CaH}_4(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$	Gram
0.378	15	0
0.756	30	0.054
1.009	40	0.080
1.512	60	0.175
2.018	80	0.545

¹ Reaction: $2.5215 \text{ grams } 3\text{CaH}_4(\text{PO}_4)_2 \cdot \text{H}_2\text{O} + 2.2460 \text{ grams } 8 \text{ NaHCO}_3 + (\text{Q.}) \text{ CaSO}_4 + 100 \text{ grams } \text{H}_2\text{O}$.

It appears that with the proportions of monocalcium phosphate and sodium bicarbonate used, the calcium sulphate was entirely decomposed when its amount as CaSO_4 was 15% of the weight of the phosphate ($\text{CaH}_4(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$).

In regard to the effect of CaSO_4 upon the production of carbon dioxide, it may be said that since both calcium sulphate and monocalcium phosphate react with sodium bicarbonate, giving off carbon dioxide, when mixed they will still react, as shown in equations 4, 5, 6, and 7 above, which in general are borne out in our experiments, results subject, of course, to variations produced by change in concentrations and temperatures.

Further, a monocalcium phosphate which has been neutralized to phenolphthalein by addition of N/10 sodium hydroxid shows a marked

¹ Storer, Am. J. Sci., 1858, **35**: (2) 41; Dulong, Ann. Chim., 1812, **82**: 273; Spiller, J. Chem. Soc., 1858, **10**: 110; Kippenberger, Z. anorg. Chem., 1894, **6**: 177.

² J. Am. Chem. Soc., 1894, **16**: 333.

increase in acidity when calcium sulphate is added. This excess acidity may be titrated with standard alkali, or it may be determined quantitatively by the electrometric method, using the hydrogen electrode to evaluate the increase in the so-called "hydrogen ion concentration." Both methods were used in this investigation.

The electrometric method has the advantage that it does not disturb the equilibrium as does the addition of alkali in titrating.

Baking experiments conducted in cooperation with Miss Hannah Wessling confirmed the conclusions from the chemical studies. Addition of calcium sulphate appreciably raised the limit of alkali which could be used with a given amount of phosphate in the baking-powder mixture without leaving an alkaline taste in the biscuit.

The experiments reported at this time have settled only the question as to whether the calcium sulphate does take part in the reaction. The exact limits of the reaction are yet to be determined.

REPORT ON MEAT AND FISH.

By E. D. CLARK (Food Research Laboratory, Philadelphia, Pa.),
Associate Referee.

Studies this year are included in two groups. The first was to determine the accuracy and general application of the Price method for starch in meat products by the analysis of known samples through the aid of the collaborators. The second group of studies included methods of detecting incipient decomposition and changes in fish flesh by determining the various forms of nitrogen in the so-called "nitrogen distribution," the changes in fat constants, etc.

PRICE METHOD FOR STARCH IN MEAT PRODUCTS.

For the last two years it has been recommended by the association that Price's method be studied further, with a view to its adoption as provisional. It seemed desirable to make a final study of this method before recommending its adoption. Previous work reported on the method shows that the directions as given are easily carried out and that theoretical values are obtained for known weights of starch added to meat. Accordingly, the immediate problem seemed to be a study of the uniformity of results obtained on the same meat sample by different analysts, rather than any further investigation of theoretical considerations.

In collaborative work of this sort on starch in meat it is impossible to prepare uniform and exact samples because of the difficulty of thoroughly mixing dry starch or cereal with a wet and greasy material like sausage meat. Accordingly, two large samples of ground meat were prepared,

using borax as a preservative. To each of these samples potato starch was added and the whole thoroughly mixed by running through a meat chopper ten times in succession. Allowing for the moisture, etc., in the starch, it was possible to prepare fairly uniform samples of chopped meat containing an approximately known weight of starch. Portions of the large samples were analyzed for starch, and then 4-ounce samples were sent out to collaborators.

In the original Price method, reduced copper is determined by titration, as proposed by Low. However, in many laboratories it is customary to determine the precipitated cuprous oxid by weighing directly, according to Munson and Walker's procedure. Therefore, in order to get comparative figures, the collaborators were requested to determine the starch in the second sample by the use of both the volumetric and gravimetric methods for reduced copper.

Results of collaborative work on the Price method for starch in meat products.

ANALYST	AVERAGES OF TWO OR MORE DETERMINATIONS			
	Sample 1 (\pm 1.30% starch)		Sample 2 (\pm 1.65% starch)	
	Volumetric	Gravimetric	Volumetric	Gravimetric
	per cent	per cent	per cent	per cent
L. H. Almy, Philadelphia.....	1.25	1.23	1.67	1.69
E. D. Clark, Philadelphia.....	1.22	1.42	1.45
R. D. Cook, Chicago.....	1.24	1.44
M. O. Johnson, Washington.....	1.33
T. R. Le Compte, Washington.....	1.67	1.65
R. M. Mehurin, Washington.....	1.57	1.53
C. G. Sutton, St. Paul.....	1.11	1.11	1.42	1.47
A. S. Thatcher, Washington.....	1.36
G. W. Trainor, Chicago.....	1.28	1.74
Average.....	1.26	1.17	1.56	1.56

A study of the collaborators' figures shows that they are in satisfactory agreement with each other and with the approximately known weight of starch added in making up the sample. The Price method has an accuracy and an ease of manipulation that make it worthy of adoption as a provisional method. To accommodate those analysts who prefer to determine the copper gravimetrically as cuprous oxid, rather than by titrating according to Low's method, the Price method should be modified to permit the weighing of cuprous oxid as an optional procedure. From our experience, and that of our collaborators, it seems that this method should be adopted as provisional in place of that of Mayrhofer.

PRELIMINARY STUDIES ON CHEMICAL METHODS OF DETECTING
DETERIORATION IN FISH FLESH.

BY E. D. CLARK AND L. H. ARMY.

The quantitative study of changes in fish flesh upon standing or during cold storage is a comparatively new one. The researches of Richardson and Scherubel, Emmett and Grindley, and others, upon meat during storage opened the way in that field. Wiley, Pennington, and collaborators, have done the fundamental work on the chemistry of decomposition processes in the flesh of poultry, game, etc. The investigations of Smith¹ and of Perlzweig and Gies² on changes in fish during cold storage are the pioneer contributions to our knowledge of the behavior of fish flesh under refrigeration.

Owing to the fact that there were few generally accepted methods for studying chemical changes in fish flesh, and also that the perishable nature of fresh fish made it impossible to send samples to collaborators, it was decided to make the preliminary studies in our own laboratory. This work was done in connection with the investigations of the laboratory on the effects of cold storage upon fish. Therefore, this report is a result of the year's experience with various methods applied to the study of fish.

The following list shows the determinations made for the tabulation of results on changes in the fish during cold storage. It was intended to include every determination that offered any possibility of giving information about fish flesh and its changes. Some of these determinations are unnecessary and some may be of little value, but all of them will help to give definite information about the adequacy of the methods themselves as applied to this problem. Most of the determinations have been adopted from the work of early investigators upon meats and poultry. The determination of the acidity of the fish extract was suggested by Smith's publication. Determination of amino acids by both the Van Slyke and Sørensen methods was a new departure. Other new methods or modifications will be apparent.

Tabulation of complete analysis of fish.

(A) SAMPLE.

- | | |
|-------------------------------------|--------------------|
| 1. Description of fish and history. | 3. Edible portion. |
| 2. Total weight. | 4. Refuse. |

(B) GROSS ANALYSIS.

- | | |
|-------------------|---|
| 1. Moisture. | 5. Total nitrogen |
| 2. Solids. | 6. Nitrogen \times 6.25 (protein). |
| 3. Ash. | 7. Water-insoluble nitrogen. |
| 4. Ether extract. | 8. Ammonia and amine nitrogen by aëra-
tion. |

¹ Smith. Biochem. Bul., 1913, 3: 54.

² Perlzweig and Gies. Biochem. Bul., 1913, 3: 69.

(C) ANALYSIS OF AQUEOUS EXTRACT.

- | | |
|--|--|
| <ol style="list-style-type: none"> 1. Acidity of extract in terms of N/10 NaOH solution with phenolphthalein. 2. Solids. 3. Ash. 4. Organic matter. 5. Nitrogen distribution in extract. <ol style="list-style-type: none"> (a) Total nitrogen of extract, or soluble nitrogen. (b) Coagulable nitrogen. | <ol style="list-style-type: none"> (c) Nitrogen precipitated by— <ol style="list-style-type: none"> (I) Tannin-salt method. (II) Zinc sulphate method. (d) Nitrogen "bases," etc., by difference. (e) "Amino-acid nitrogen" by— <ol style="list-style-type: none"> (I) Van Slyke method. (II) Sørensen titration method. (f) Ammonia and amine nitrogen by aëration. |
|--|--|

(D) STUDY OF FAT EXTRACTED FROM FLESH.

- | | |
|---|---|
| <ol style="list-style-type: none"> 1. Specific gravity. 2. Index of refraction. 3. Iodin number. | <ol style="list-style-type: none"> 4. Saponification number. 5. Acid number. 6. Acetyl number. |
|---|---|

There is no need to discuss the ordinary determinations like total nitrogen, ash, fat constants, etc., but in the case of ammonia nitrogen there are certain complications that must be considered. In the determination of the so-called ammonia nitrogen we used a modification of the Folin aëration method. We followed Smith in using the Steel¹ modification of Folin's method because Perlzweig and Gies reported the presence of crystals of ammonium magnesium phosphate in fish flesh even early in the process of deterioration. The use of sodium hydroxid and sodium chlorid in place of the sodium carbonate, as suggested by Steel, seems to be necessary in cases like the analysis of fish where magnesium ammonium phosphate is known to be present. Benedict and Osterberg² have made a detailed study of the use of Steel's modification as well as the original Folin method upon urines, and they report that the former method apparently gives correct results for ammonia under nearly every condition. In fish flesh not only do we have the interference of this magnesium compound but also the added complication of amines, unknown as to nature and amount. Later we will show that certain classes of amines may be determined quantitatively by the aëration method, either when alone or with ammonium salts. Since this is true, and since we know that amines occur in fish, we found it desirable to use Steel's modification, and later to make a study of its efficiency in determining amines, either in the presence or absence of ammonium salts.

In order to study the interference, if any, of the amines with the determination of ammonia, we carried out the following experiments: Standard solutions of methyl amine, trimethyl amine, and ammonium chlorid were prepared. The exact amount of nitrogen contained in 5 cc. portions of these solutions was ascertained by duplicate Kjeldahl deter-

¹ Steel. J. Biol. Chem., 1910, 8: 365.

² Benedict and Osterberg. Biochem. Bul., 1913, 3: 47.

minations. Then the same amounts of these standard solutions were analyzed by the Folin aëration method, using Steel's modification, as already noted. These solutions were analyzed separately and mixed in stated proportions. The figures by the aëration method are the averages of four separate determinations.

Determination of ammonia and amine nitrogen by the aëration method.

SUBSTANCE USED	NITROGEN BY AÉRATION	NITROGEN BY KJELDAHL METHOD
	gram	gram
Methyl amine solution alone.....	0.0052	0.0050
Trimethyl amine solution alone.....	0.0072	0.0077
Ammonium chlorid solution alone.....	0.0189	0.0188
Trimethyl amine + NH_4Cl , solutions mixed.....	0.0148	0.0143
Trimethyl amine + CH_3NH_2 , solutions mixed.....	0.0124	0.0127

It is evident from these results that the primary or tertiary amines, either alone or with ammonium salts, are determined quantitatively by the modified Folin method for ammonium nitrogen. Therefore, we consider that determinations by this method give "ammonia and amine" nitrogen. Proteins do not interfere.

We already have seen that the presence of amines and magnesium compounds in fish is a disturbing factor in the determination of the so-called "ammonia" nitrogen by the aëration method. In the past it usually has been customary to study the amino-acid content of flesh extracts when inquiring into the effect of cold storage upon such material. In our own work we used the Sørensen¹ titration method for amino acids in making parallel determinations along with the gasometric method of Van Slyke.² In determining amino acids in fish flesh, account must be taken of the presence of both ammonia and amines, which are very closely related to the amino acids, and have many properties in common with them. For this reason a study was made of the effect of ammonia and amines, separately and in combination, upon the Sørensen titration.

Our study of the behavior of ammonia and amines in amino-acid titrations was carried out in much the same way as that upon the aëration method for ammonia described above. Standard solutions of leucin, of ammonia, and of different amines were prepared and analyzed for their "amino-acid nitrogen" according to the Sørensen method. Later, definite volumes of these same solutions were mixed and then analyzed for amino-acid nitrogen in the same way. It is obvious that if there were no interference between the ammonia, amines, and amino acids the results of the titrations upon the mixed solutions would be the same as those upon

¹ Sørensen. *Biochem. Z.*, 1907-8, 7: 44.

² Van Slyke. *J. Biol. Chem.*, 1912, 12: 275, and *idem.*, 1913, 16: 121.

the solutions analyzed separately. Following are some results of this work:

Effect of ammonium chlorid and amines upon the determination of amino-acid nitrogen by the Sørensen method.

SUBSTANCES USED	NITROGEN DETERMINED SEPARATELY	NITROGEN DETERMINED IN MIXTURE
	gram	gram
Leucin solution alone.....	0.0059
Methyl amine solution alone.....	0.0072
Trimethyl amine solution alone.....	0.0017
Ammonium chlorid solution alone.....	0.0089
Leucin + NH_4Cl , solutions mixed.....	0.0148	0.0148
Leucin + CH_3NH_2 , solutions mixed.....	0.0131	0.0133
Leucin + $(\text{CH}_3)_3\text{N}$, solutions mixed.....	0.0076	0.0076
Leucin + CH_3NH_2 + NH_4Cl , solutions mixed.....	0.0219	0.0215
Leucin + $(\text{CH}_3)_3\text{N}$ + NH_4Cl , solutions mixed.....	0.0165	0.0159

An inspection of this table shows that the presence of ammonia in relatively small quantities does not seriously interfere with the amino-acid determination; neither does the presence of amines; but when amines and ammonia occur together the results obtained by the Sørensen titration are not reliable. Unfortunately, this is exactly the state of affairs existing in fish flesh. This work upon the interference of certain nitrogen compounds with the Sørensen amino-acid determination is a confirmation of the observations made by Henriques and Sørensen¹ upon the disturbing influence of ammonium salts and methyl amine. It is evident, then, that the determination of amino acids by the above method gives results that are inaccurate owing to the interference on the part of ammonia and amines. This interference, unfortunately, is not a definite one, and for that reason it is not allowable to correct the amino-acid results by subtracting the amount of nitrogen present as ammonia plus amines, determined by the aëration method. Therefore, in all the tables the percentage of amino acid as reported is subject to a varying and unknown correction, due to the presence of ammonia and amines in the fish flesh.

It is well known to organic chemists that various types of amines react in different and characteristic ways with nitrous acid; therefore, the Van Slyke gasometric method for amino acids is likewise subject to correction on account of the presence of various types of amines in fish flesh. If the time allowed for the reaction between the nitrous acid and the substance being analyzed is sufficiently long we know that the ammonia will be determined quantitatively by the Van Slyke method. However, it is not customary to allow the reaction to run more than three or five minutes. For that reason our results for amino acids determined by

¹ Henriques and Sørensen. Z. physiol. Chem., 1910, 64: 120.

the Van Slyke method are also subject to correction by an unknown amount, due to the presence in fish flesh of different types of amines and ammonia, and must not be viewed as giving accurate information on the percentage of amino acids alone.

To make the Sørensen and Van Slyke determinations of amino acids entirely accurate, it would, of course, have been possible to remove the ammonia and amines by distilling with calcium hydroxid according to Van Slyke's procedure. But this precaution seems hardly necessary in our work where the quantities of amino acids and ammonia are about 0.15% and 0.01%, respectively. Furthermore, the comparative value of our uncorrected amino-acid figures on extracts of fish flesh in studies of nitrogen distribution are not impaired, because the determinations are always made in exactly the same way on nearly identical material at definite periods during two years.

Many parallel determinations were made of nitrogen precipitated by the tannin-salt reagent and by zinc sulphate applied to the fish extracts. The results obtained by the latter method were constantly lower than those from the tannin-salt method. It is known that the zinc sulphate precipitates most sharply those substances commonly classed as proteoses, while the other method includes certain other nitrogenous substances as well as proteoses. However, in our work we always use both methods in order to have comparative figures running through the whole period of cold storage. We have found it very convenient and accurate to filter off the zinc sulphate precipitate on an asbestos mat in a Gooch crucible, wash mat and precipitate into a Kjeldahl flask and transfer for the nitrogen determinations.

In the space at our disposal it is impossible to indicate the details of analytical procedures used in our various determinations, as listed on pages 231-232. The strict precautions to be followed in preparing aqueous extracts of fish flesh, preserving them, taking aliquots, etc., may also be omitted at this time. This is also true of the details of extracting and analyzing the oils from the fish. In another year the results of all our work on frozen fish stored for two years will be completed and tabulated. At that time the comparative value of all the different determinations in showing changes in fish flesh will become evident and may be discussed more intelligently.

It is obvious that all of the methods so far applied in our work on fish aim to detect deterioration by changes in constituents *already present*, caused either by the action of bacteria or enzymes in the fish itself. For many purposes it seems very desirable to find methods that will show deterioration by the detection of decomposition products *not found* in perfectly fresh fish. We have in mind such things as indol, skatol, phenolic substances, and similar products of protein decomposition. The evidence

of the early stages of decomposition in fish is not easy to get, and no stone should be left unturned in a search for indications of the tainted condition in this class of foods.

RECOMMENDATIONS.

It is recommended—

(1) That Price's method (U. S. Bur. Chem. Circ. 108, p. 10) be made a provisional method for starch in meat products in place of Mayrhofer's method (U. S. Bur. Chem. Bul. 107 (rev.), p. 109, (b) (2)). With very little modification from Price's description, the official description should be:

In a 200 cc. beaker treat 10 grams of finely divided meat with 75 cc. of an 8% solution of potassium hydrate in 95% alcohol, and heat on the steam bath until all the meat is dissolved. This will require from 30 to 45 minutes. Add an equal volume of 95% alcohol, cool, and allow to stand at least one hour. Filter by suction through a thin layer of asbestos in a Gooch crucible. Wash twice with warm 4% potassium hydrate in 50% alcohol and then twice with warm 50% alcohol. Discard the washings. Endeavor to retain as much of the precipitate in the beaker as possible until the last washing. Place the crucible with contents in the original beaker, add 40 cc. of water and then 25 cc. of concentrated sulphuric acid. Stir during the addition of the acid and see that the acid comes in contact with all the precipitate. Allow to stand about 5 minutes, add 40 cc. of water, and heat just to boiling, stirring constantly. Transfer the solution to a 500 cc. graduated flask, add 2 cc. of a 20% aqueous solution of phosphotungstic acid, allow to cool to room temperature, and make up to mark with distilled water. Filter through a starch-free filter paper, and determine the dextrose present in a 50 cc. portion of the filtrate with Fehling's solution after neutralizing the acid, using Low's method, as given in U. S. Bureau of Chemistry Bulletin 107 (revised), page 241, for the determination of the copper in the cuprous oxid precipitate, or the latter may be weighed as directed on page 242. The amount of dextrose multiplied by 0.9 gives the equivalent in starch.

(2) That comparative studies be continued of the different determinations of the nitrogen distribution and the best manner of carrying them out as applied to extracts of fish flesh. Efforts should be made to standardize the method of determining coagulable proteins in such work. New methods proposed for the determination of proteoses, amino acids, amines, etc., in biological material should be investigated with a view to finding more sensitive and more accurate indices of deterioration in fish.

(3) That a study be made of the value of volatile distillation products like amines, indol, etc., in detecting incipient decomposition in fish.

REPORT ON FATS AND OILS.

By R. H. KERR (Bureau of Animal Industry, Washington, D. C.),
Associate Referee.

The attention of the associate referee on fats and oils has been given to a careful consideration of some of the new problems which have been raised by the rapid increase in the commercial practice of hardening oils by hydrogen. While the practice so far has been applied mainly to cottonseed oil, almost any edible oil may have its value so enhanced by this treatment as to make its hydrogenation a source of profit. It must be assumed, then, that the problems of those unlucky chemists who are responsible for detections of the adulteration of edible oils and fats are now to be increased and complicated by the addition to the list of possible adulterants of a long list of hydrogenated and partly hydrogenated fats. Aside from the evident fact that the would-be adulterator may now alter at will the iodine number, refractive index, and titer of any fat he may choose, the process of hydrogenation brings about other changes which are of far-reaching importance. The Halphen test of cottonseed oil disappears, the erucic acid of rape and mustard oils becomes behenic acid, the highly unsaturated acids of the fish oils are reduced to solid acids which no longer give the characteristic precipitate of insoluble bromids. It is to be seen at once that many of our tried and trusty tests will fail us completely before this new problem, and it is equally plain that we must devise new tests to meet the new condition or confess failure.

One of the most striking features of the problem is the absolute lack of definite information regarding the composition of fats. Although fats have been studied and worked over by chemists since the time of Chevreul, and the literature of the subject comprises a large number of bulky volumes, it remains true today that we do not know accurately the composition of a single fat. We know that cottonseed oil, for example, consists of the glycerids of palmitic, oleic, and linolic acids, but we do not know whether it consists of a simple mixture of the three triglycerids of these acids, or whether the whole array of possible mixed glycerids may not also be present. In fact, I am not aware that one single glycerid ever has been isolated from cottonseed oil and identified. Almost as much may be said of many other well-known fats. It was not known until two years ago that lard did not contain the glycerid tristearin despite all the thousands of analyses of lard made every year.

In dealing with the problem raised by the use of hydrogenated fats, it appears to me that the first requirement will be a much more extensive and intimate knowledge of the exact composition of fats. When we know of what glycerids a fat is composed we are then in a position to recognize

that fat wherever we find it, tedious and difficult as the process of identification may be. Accurate knowledge may, however, point the way to short and simple tests, just as Boemer's tedious fractional crystallization of lard finally led him to a method only a little less simple and speedy than the method already adopted by this association for the detection of the adulteration of lard. A modification of this method which I hope to present to this association at a later meeting will set the detection of beef fat, mutton fat, and all of the hydrogenated oils in lard upon a firm foundation and also will enable us to distinguish between natural lard and lard hardened by addition of hydrogen. The method, if presented, will be offered as a method for the detection of the glycerid tristearin in lard, not as a method for the detection of beef fat.

Our needs appear, then, to be (1) the collection of a large amount of wholly new information, and (2) new and less laborious methods whereby the needful knowledge may be acquired. It is along the second line that I have been working. The work I have been doing has not yet reached the stage of definite methods of analysis available for coöperative study, but some progress has been made along that line, and some improved methods probably will be available for study during the coming year. The ones suggested at present are:

- (1) A better method for the detection and identification of tristearin.
- (2) A better method for the separation of solid and liquid fatty acids.
- (3) A method for the separation of the liquid fatty acids from each other.
- (4) A method for the detection of cholesterol in mixture of animal and vegetable fats.

It is hoped that at least one, and possibly two, of these methods may be studied coöperatively and the results of the study presented at the next meeting of the association.

Mr. Hand at this point explained the operation of an apparatus for the extraction of fats which he exhibited.

REPORT ON DAIRY PRODUCTS (ADULTERATION).

BY JULIUS HORTVET (State Dairy and Food Department, St. Paul, Minn.), *Associate Referee*.

The work of the past two years has magnified the importance of a special study of methods for the determination of fat in dairy products. It has, in fact, been realized more than heretofore that we are in urgent need of a thorough investigation of methods which have been recommended for study by this association; and, having in view the impor-

tance of advancing our work from year to year along the lines indicated by these recommendations, attention is first directed to the committee reports on this subject since 1906:

(1) At the meeting held in November, 1906, it was recommended, "That the Gottlieb method (Landw. Vers.-Sta., 1892, 40: 1) be made provisional for the determination of fat in milk. Referred to referee for 1907 for further recommendation. Adopted."¹

(2) At the meeting held in October, 1907, it was recommended that the study of methods of analysis of condensed milk be continued and, among other things, special attention be given to the "Roese-Gottlieb method for the determination of fat as described in the proceedings of the association for 1906. Adopted."²

(3) At the meeting held in November, 1908, it was recommended, "That the determination of fat in condensed milk be studied, special attention being given to solutions of less than 20% concentration."³

(4) At the meeting held in August, 1909, it was recommended, "That methods for determining fat in both sweetened and unsweetened condensed milk be studied, giving special attention to those modifications of the Babcock and extraction methods recently brought out in Bulletin 134 of the Indiana Agricultural Experiment Station. Adopted."⁴

(5) At the meeting held in November, 1910, it was recommended, "(1) That the Roese-Gottlieb method be adopted as provisional for the determination of fat in milks and condensed milk, both sweetened and unsweetened. Carried. (Recommended to the committee by G. E. Patrick as official.) (2) That the Roese-Gottlieb method be further studied for the analysis of ice cream, milk powders, malted milks, and milk chocolates. Adopted."⁵

(6) At the meeting held in November, 1911, it was recommended, "That Paul's method of extracting fat from dairy products be further studied, and that the fat obtained by this method be studied as to its chemical and physical constants. Adopted."⁶

During the meetings held in 1912, 1913, and 1914, the reports of the associate referee on dairy products were devoted chiefly to a continuation of the study of Paul's continuous-extraction method, together with the modifications of the method which have been proposed. Examination of the work on dairy products during the past eight years reveals the fact that the recommendations adopted during that period have not been consistently carried out. In fact, so far as the proceedings of the

¹ U. S. Bur. Chem. Bul. 105, p. 154.

² Ibid., 116, p. 117.

³ Ibid., 122, p. 188.

⁴ Ibid., 132, p. 188.

⁵ Ibid., 137, p. 169.

⁶ Ibid., 152, p. 188.

past eight years indicate, the Roesse-Gottlieb method has been adopted only as provisional. It appears to have been recommended for adoption as official, but, so far as can be learned, no further definite action along that line has been taken. Also, the further recommendations for study of the Roesse-Gottlieb method and other methods for the determination of fat in ice cream, milk powders, malted milk, etc., appear to have been overlooked. It therefore has been the aim of the associate referee during the past season to take up the study of methods for determining fat in dairy products along the lines recommended by this association between the years 1906 and 1911. The work on Paul's continuous-extraction method was practically disposed of at the meeting held in November, 1914, and no definite recommendation for the final adoption of that method was made, although it appeared that the method was valuable and serviceable in certain cases and for the special purpose for which it was originally designed. Taking up the work on dairy products at the stage indicated in the proceedings of 1911, the associate referee has accordingly enlisted the assistance of about twenty collaborators in a further study of the Roesse-Gottlieb method, with a view to placing this method in line for final adoption as official in 1916. Also, following out the instructions of this association as implied in the recommendations adopted at the meetings held in 1909 and 1910, the plan of work of the past season has included a comparative study of several modifications of the Babcock method which have been proposed during recent years. The following instructions were sent out to the collaborators:

INSTRUCTIONS TO COLLABORATORS.

Herewith I am sending you directions for collaborative work on dairy products for 1915. These directions include a description of various methods which have been proposed for the determination of fat in evaporated milk and ice cream. Also there is included a description of the Roesse-Gottlieb method, together with general instructions. You are requested to make a comparative study of all of these methods, and for that purpose to obtain in the local market two samples of each of the products named in the directions, viz., *whole milk*, *sweet cream*, *evaporated milk*, and *ice cream*. Instead of attempting to send out uniform samples to the collaborators, the above plan has been decided upon as preferable. Each collaborator will then carry out his work on samples obtained by himself, and, in addition, if time permits, may extend the work on as many additional samples as he may wish to investigate. You are requested to report your results in the plan shown in the tabular form herewith inclosed, and to write out in full your discussions and criticisms on the various methods.

During recent years a number of methods have been devised for testing butter-fat in dairy products, and a considerable amount of uncertainty has arisen regarding the reliability of standard methods heretofore employed. It therefore has been deemed advisable to subject several of the most important of these tests to a careful critical study alongside of the well-tried-out Roesse-Gottlieb procedure adopted as provisional a few years ago. There is an urgent call for a reliable centrifugal

method for condensed milk and ice cream, and it is hoped that the collaborative work of the present season will bring about some agreement, at least on essential points, in connection with one or more of the methods which have been proposed.

Later in the season, as a result of correspondence with Dr. W. D. Bigelow, chief chemist of the National Canners' Association Laboratory, it was decided to supplement the work outlined in these instructions by determinations to be carried out on specially prepared samples of evaporated and condensed milk. These samples were put up under the careful supervision of Dr. Bigelow, at the factory of Borden's Condensed Milk Company, at Norwich, N. Y., and sent out direct to the collaborators. The samples included: (1) An unsweetened evaporated milk, (2) a sweetened condensed milk, (3) a sweetened condensed milk of composition different from sample 2. In connection with these samples, the following additional instructions were sent to the collaborators:

In addition to the work outlined in the instructions which I sent you some time ago, I will request that you make determinations of butterfat on these three samples by the Roesse-Gottlieb method, and, in addition, by any other method which you believe may give correct results. You may select any one of the methods described in the outline sent you; or if you have another method which you have employed in your laboratory, I should be pleased to have you report results by that method. Results obtained on these samples are to be reported at the same time that you send me your statement covering the work included in my outline already sent you.

Carrying out the plan of work as originally outlined in the first letter of instructions, the collaborators were given the following description of methods:

DESCRIPTION OF METHODS.

BRINSMAID METHOD.

Place 2 cc. of glycerin in a Babcock milk test bottle and rotate so that the lower half surface is coated with the glycerin. In a small beaker mix 12 cc. of sulphuric acid (C. P., sp. gr. 1.84) with 18 cc. of 80% acetic acid. Weigh 9 grams of the well-mixed sample into the bottle and pour in the mixed acids so that the neck of the bottle is well washed down, then rotate the bottle so that the milk and acids are mixed and all white lumps dissolved. After thorough mixing, set the bottle aside for 15 minutes, the mixture in the meantime becoming black as in the ordinary Babcock test. Arrange a suitable rack or support so that the bottle may be placed standing in boiling water, allowing water of sufficient depth to cover the bottle above its contents. Leave the bottle in the water for 20 minutes, mixing the contents several times during this period, run in the centrifuge 5 minutes, add hot water so that the contents of the bottle rise to the bottom of the neck, then run 2 minutes, add hot water until the fat rises to the upper part of the neck, and complete the test by making a final run of 1 minute. Measure the fat column and express the result in the manner described under the heading, "Reading the fat column."

Notes on the method.—The preliminary coating of the inside of the bottle with glycerin to some extent prevents the milk from adhering to the sides. A good

method of introducing the glycerin into the bottle is to take an ordinary 2 cc. pipette, cut off the tapering point, draw the glycerin into the pipette, wipe off the outside around the end, place the pipette down into the bottle, and allow the glycerin to drain out. This procedure will prevent the glycerin from coming in contact with the neck of the bottle. Considerable heat is developed when the acids are mixed, but there is no danger of the mixture becoming too hot. A thorough mixing of the milk and acids is necessary for good results. This method appears to have been in use for several years in the laboratory of the Illinois Dairy and Food Commission. Mr. David Klein, Illinois State analyst, proposes that in the case of condensed milk a correction figure of 0.24 be subtracted from the result after multiplying the reading by 2.

GRIGSBY METHOD.

Weigh 9 grams of the thoroughly mixed sample into a Babcock milk test bottle and add 10 cc. of glacial acetic acid, washing down the neck of the bottle with the acid, then heat the mixture by putting the body of the bottle in hot water until the contents appear smooth, uniform, and free from lumps. While hot, add ordinary Babcock sulphuric acid, about 1 or 2 cc. at a time, mixing the contents after each addition, until the color of the mixture becomes dark chocolate brown, centrifuge 5 minutes, run in hot water to the bottom of the neck, centrifuge 2 minutes longer, then run in hot water until the fat column comes nearly to the top of the neck, and complete with a final run in the centrifuge of 1 minute. Measure the fat column and express the result as directed in the paragraph headed, "Reading the fat column."

Notes on the method.—Too long heating or overheating of the mixture of acetic acid and sample has practically no effect on the result, heating for 5 to 10 minutes in water slightly below boiling temperature being usually sufficient. The hotter the mixture the less sulphuric acid will ordinarily be required to produce the proper result. The fat column should be perfectly clear and free from curd or char. In case either curd or char appears, the test should be repeated, adding sulphuric acid so as to produce a darker color in the case of interfering curd or so as to produce a lighter color in the case of interfering char.

WENDLER METHOD¹.

Prepare a reagent having the following composition: 125 grams sodium hydroxide, 25 grams Rochelle salt, 25 grams sodium chloride; dissolve in water, mix the solution, and make volume up to 500 cc. In testing whole milk, run 19 cc. of the reagent into a Babcock test bottle, add 17.6 cc. of the sample in such manner as to float over the reagent, then add 10 or 12 drops of isobutyl alcohol. In testing cream, weigh 18 grams of the sample into the test bottle, run in 19 cc. of the reagent so as to displace the cream, then add 10 or 12 drops of isobutyl alcohol. In testing evaporated milk, weigh 9 grams of the sample in the bottle, add 9 cc. of water and thoroughly mix, then run in 19 cc. of reagent so as to displace the sample, and add 10 or 12 drops of isobutyl alcohol. In testing ice cream proceed as in the case of evaporated milk. In each case, after introducing sample and reagents, shake the mixture for several minutes, place the test bottle in a water bath at 50°C. for 3 to 5 minutes in the case of milk and cream, and in the case of evaporated milk and ice cream heat the mixture at 60°C. 5 to 7 minutes. Shake thoroughly during the heating, then centrifuge at highest possible speed for 4 minutes. Add boiling hot water to bring the fat column up to the graduations in the

¹ Z. öfent. Chem., 1906, 12: 41-58.

neck, centrifuge an additional minute, measure the fat column, and express results as directed in paragraph headed, "Reading the fat column."

In addition to the methods to be subjected to comparative study described above, determine fat in each sample according to the Roese-Gottlieb method. Also, in the case of milk and cream samples make the fat determination according to the Babcock method carried out according to procedure commonly recognized as correct.

For convenience in making comparative tests by the Roese-Gottlieb method, the following description is added.

ROESE-GOTTLIEB METHOD.

Weigh 40 grams of the properly prepared sample, preferably in a tared weighing dish used for sugar analysis, transfer by washing to a 100 cc. graduated sugar flask, and make up to the mark with water. Measure 10 cc. of this solution into a Röhrig tube or into a suitable size Werner-Schmidt extraction apparatus, using for the purpose not more than 10 cc. of water to transfer sample to the tube. To the material in the extraction tube add 1.25 cc. of concentrated ammonium hydroxid (2 cc. if the sample be sour) and mix thoroughly. Add 10 cc. of 95% alcohol and mix well, then add 25 cc. of washed ethyl ether, shake vigorously for half a minute, add 25 cc. of petroleum ether (redistilled slowly at a temperature below 60° preferably) and shake for half a minute. Let stand 20 minutes, or until the upper liquid is practically clear and its lower level constant. Draw off the ether-fat solution as much as possible (usually 0.5 to 0.9 will be left) into a weighed flask through a small quick-acting filter. Re-extract the liquid remaining in the tube, this time with only 15 cc. of each ether, shaking vigorously half a minute, and allow to settle. Draw off the clear solution through the small filter into the same flask as before and wash the tip of the outlet, the funnel, and filter with a few cc. of a mixture of the two ethers in equal parts. Extract again and wash in the manner just described. Evaporate the ether slowly on a steam bath, then dry the fat in a water-oven until loss of weight ceases.

Notes on the method.—Cream and evaporated milk are best sampled by taking 40 grams and dilute to 100 cc. A 10 cc. sample of this mixture is taken for analysis. In the case of ice cream or other products which are not smooth and which contain air bubbles, 4 grams of the thoroughly mixed sample are weighed and introduced into a Röhrig tube with the aid of 10 cc. of water, then ammonia, alcohol, and ether are added in the amounts called for by the method. In the case of whole milk it has been found convenient to weigh out 10 grams directly. If an emulsion occurs in the Röhrig tube the addition of a little alcohol followed by shaking will usually break it up. Also, the use of a tube of larger caliber will tend to overcome this difficulty. Or, use a large test tube provided with a blowing-off device, as in the Werner-Schmidt method. Care should be taken to weigh the residue after evaporation of ether under exactly the same conditions as those under which the dish was weighed. If the fat contains casein or foreign matter, re-extract with ether, evaporate, and weigh again. If all proper precautions are taken, results on a given sample should check within 0.02 to 0.05%. If the result is not satisfactory repeat the analysis.

GENERAL INSTRUCTIONS.

(1) *Condition of samples.*—All samples should be in the best condition possible. An evaporated milk should be perfectly smooth and contain no small lumps of fat or curded matter.

(2) *Sampling ice cream.*—Allow the sample to soften at room temperature, with frequent stirring with a spoon. A good method of obtaining a uniform sample is

to mix with an egg beater just before weighing. It has been found difficult to obtain a fair sample after the ice cream has been heated on the water bath, owing to the fact that the butterfat is melted and quickly rises to the surface.

(3) *Babcock test bottle*.—Use an 18-gram milk test bottle, reading to 8% and graduated to 0.1 of 1%.

(4) *Reading the fat column*.—Measure with a pair of dividers and read (a) from the extreme bottom to the extreme top, (b) from the extreme bottom to the lower line of the meniscus. Record and report both results. In cases where a 9-gram sample has been taken for the test, add the two readings (a) and (b) to obtain the percentage of fat.

(5) *Experience*.—In the case of all methods make preliminary trials. Repeat as many times as seem necessary in order to become adequately prepared for the regular determinations to be made on selected samples. The importance of ample experience cannot be overestimated, especially in connection with the *Rosse-Gottlieb* method.

(6) *Plan of work*.—The following outline will serve to indicate the determinations which are to be made on the various samples named in the heading.

MILK	CREAM	EVAPORATED MILK	ICE CREAM
Rosse-Gottlieb Wendler Babcock	Rosse-Gottlieb Wendler Babcock	Rosse-Gottlieb Wendler Brinsmaid	Rosse-Gottlieb Wendler Grigsby

The results reported by the collaborators have been compiled and arranged for purposes of comparison as shown in the following tables.

The results shown in Tables 1 and 2 indicate chiefly a comparison of the Wendler and Brinsmaid methods with the *Rosse-Gottlieb* as described in the instructions to the collaborators. In methods directing the use of a 9-gram sample of evaporated or condensed milk the collaborators were instructed to compute the fat percentages by three different formulas. A comparison of the results reported by these methods indicates that the sum of the readings designated (a) and (b) represents a fair basis on which to make the comparisons. The results obtained by multiplying reading (a) by 2 and deducting 0.24 were for the most part apparently too low. This method of expressing the fat measurement was, however, originally intended for tests applied to ice cream. With very few exceptions, the results obtained by multiplying reading (b) by 2 and adding 0.15 agreed substantially with the results obtained by adding fat-column readings (a) and (b). It therefore has been decided, for purposes of comparison of results by the various methods, to confine the Wendler and Brinsmaid figures to the sum of readings (a) and (b). Results by various miscellaneous methods are also shown in the tabulations.

Mr. J. T. Keister, of the dairy laboratory, U. S. Bureau of Chemistry, has made a special study of the methods which were described in the instructions to the collaborators. These methods have been subjected to a large number of comparative tests on samples of milk, cream, evaporated

TABLE 1.
Fat determinations in condensed milk.
 COLLABORATORS' SAMPLE No. 1—EVAPORATED MILK.

ANALYST	ROSE- GOTTLIEB	WENDLER	BRINEMAID	OTHER METHODS
H. B. Burnett.....	8.05	8.10	8.20
R. E. Stevenson.....	8.06	7.65	7.85
C. Bahlman.....	8.17	8.05	8.15
C. G. Sutton.....	7.98
	7.96	7.95	8.15
	7.93	7.96	8.15
	7.94
G. B. Taylor.....	7.60
	7.56	7.85
H. S. Bailey.....	8.06
E. W. Thornton.....	8.01	8.10	7.90
C. L. Black.....	7.63	7.95
	7.66	8.10
	7.81	8.21	8.14	*7.58
	7.81	8.12	8.13	*7.40
C. N. Austin.....	7.83	8.16	8.12	*7.62
	7.82	8.14	*7.78
	7.81	*7.54
	*7.32
W. D. Strack.....	7.98
	7.97	*8.3
	7.98	*8.3
J. H. Bornmann.....	7.95	8.00	*8.05
	7.92	7.95	*7.96
F. C. Broeman.....	7.76	7.85	7.95
	7.98
	7.97
	7.96
F. F. Fitzgerald.....	7.96
	7.96
	7.98
	7.99
H. M. Miller.....	8.00
	8.03
	7.96
P. J. Donk.....	7.97
	7.97
	7.94
Indiana Condensed Milk Co...	7.98
	7.98
W. C. Geagley.....	7.97	6.8	8.0	*8.0
	8.00	5.8	8.0	*8.0
	7.97
C. L. Munroe.....	7.94
	7.86
	7.90
H. E. Otting.....	8.20
	8.10	8.10	*8.60
H. A. Halvorson.....	7.94
	7.89
J. T. Keister.....	8.05	*8.08
	8.06	*7.96
O. L. Evenson.....	*8.02
	8.01	*8.05
Maximum.....	8.10
Minimum.....	7.81
Average.....	7.96

¹ Not included in calculations.
² Grigsby method.
³ Beimling method.

⁴ Hunsiker method.
⁵ Biesterfeld-Evenson method.

TABLE 1.—Continued.
COLLABORATORS' SAMPLE No. 2—SWEETENED CONDENSED MILK.

ANALYST	ROMBE- GOTTLIEN	GRIGSBY	BRINE- MAID	WENDLER	OTHER METHODS
L. B. Burnett.....	9.05	9.05			
R. E. Stevenson.....	9.13	9.30			
C. Bahlman.....	9.07		8.35	8.20	
C. G. Sutton.....	9.10				
	9.13				
G. B. Taylor.....	8.96				
H. S. Bailey.....	9.00				
E. W. Thornton.....	¹⁹ 9.38				
C. L. Black.....	¹⁹ 9.49				
	¹⁸ 8.57				
	9.14	8.80	9.20		
C. N. Austin.....	9.10	8.92	9.11		
	9.11	8.81	9.18		
		9.04	9.24		
	9.09				
W. D. Strack.....	9.08				¹⁹ 9.2
	9.07				¹⁹ 9.1
J. H. Bornmann.....	9.17				¹⁸ 8.1
	9.12				¹⁸ 8.4
E. C. Broeman.....	¹⁷ 9.95				
F. F. Fitzgerald.....	9.07				
	9.09				
H. M. Miller.....	9.09				
	9.08				
	9.07				
P. J. Donk.....	9.08				
C. L. Monroe.....	¹⁹ 9.58				
	¹⁸ 8.62				
J. T. Keister.....	¹⁹ 9.24				¹⁹ 9.20
	¹⁹ 9.245				¹⁹ 9.25
	9.12				¹⁹ 9.12
O. L. Evenson.....	9.05				¹⁹ 9.12
H. A. Halvorson.....	9.08				
	8.99				
Maximum.....	9.17				
Minimum.....	8.96				
Average.....	9.08				

¹ Not included in calculations.
² Beimling method.

³ Leach method.
⁴ Biesterfeld-Evenson method.

TABLE 1.—Continued.

COLLABORATORS' SAMPLE No. 53N8A—SWEETENED CONDENSED MILK.

ANALYST	ROSE- GOTTLIEB	GRIGSBY	BRINSMAID	OTHER METHODS
L. B. Burnett.....	8.95	9.20
R. E. Stevenson.....	8.15	8.80
C. G. Sutton.....	8.86	8.85
G. B. Taylor.....	8.84	8.92
H. S. Bailey.....	8.85
E. W. Thornton.....	8.99
C. L. Black.....	8.60
	8.06
	8.04	9.21
C. N. Austin.....	8.90	8.52	9.12
	8.90	8.59	9.14
	8.91	8.81	9.24
	8.83
W. D. Strack.....	8.83	9.10
	8.84	9.00
J. H. Bornmann.....	8.89	8.55
F. C. Broeman.....	8.86	8.40
F. F. Fitzgerald.....	7.38
	8.97
	8.95
	8.95
H. M. Miller.....	8.96
	8.93
	8.97
P. J. Donk.....	8.93
	8.93
C. L. Munroe.....	8.58
	8.62
J. T. Keister.....	9.105	9.116
	9.13	9.075
O. L. Evenson.....	8.95	9.00
	8.98	9.05
H. A. Halvorson.....	8.93
	8.96
	8.81	6.8	6.2
W. C. Geagley.....	8.82	6.8	5.9
	8.83	7.2
	7.2
Maximum.....	8.99
Minimum.....	8.81
Average.....	8.90

¹ Average of the following determinations: 8.93, 8.97, 8.94, 8.96, 8.96, 8.94.² Not included in calculations.³ Biesterfeld-Evenson method.⁴ Beilmling method.⁵ Leach method.⁶ Wendler method.⁷ Hunsiker method.

TABLE 2.
Collaborative fat determinations in milk, cream, and ice cream.
MISCELLANEOUS MILKS.

ANALYST	ROSE- GOTTLIEB	WENDLER	BABCOCK	VARIATIONS FROM ROSE-GOTTLIEB	
		Average of (a) and (b) readings	Average of (a) and (b) readings	Wendler	Babcock
H. B. Burnett.....	3.70	3.69	3.52	-0.01	-0.18
	3.79	3.75	3.52	-0.04	-0.27
R. E. Stevenson.....	3.70	3.60	3.40	-0.10	-0.30
	3.62	3.65	3.45	+0.03	-0.17
	3.56	3.68	3.60	+0.12	+0.04
C. Bahlman.....	4.69	4.78	4.80	+0.09	+0.11
C. G. Sutton.....	¹ 3.96	¹ 4.09	¹ 4.00	+0.13	+0.04
G. B. Taylor.....	3.78	3.65	3.80	-0.13	+0.02
E. W. Thornton.....	4.58	4.58	4.53	0.00	-0.05
C. L. Black.....	¹ 3.18	¹ 3.05	¹ 3.23	-0.13	+0.05
	¹ 4.18	¹ 4.04	¹ 4.28	-0.14	+0.10
J. H. Bornmann.....	¹ 3.32	¹ 3.33	3.25	+0.01	-0.08
	¹ 3.56	¹ 3.28	3.55	-0.28	-0.01
H. A. Halvorson.....	4.61	4.65	4.55	+0.04	-0.06
W. C. Geagley.....	3.06	3.05	3.00	-0.01	-0.06
	3.45	3.40	3.43	-0.05	-0.02

MISCELLANEOUS CREAMS.

H. B. Burnett.....	18.25	18.00	17.60	-0.25	-0.65
	18.37	18.80	17.60	+0.43	-1.20
R. E. Stevenson.....	18.05	18.40	17.48	+0.35	-0.57
	18.45	19.20	18.40	+0.75	-0.05
	17.69	17.88	18.50	+0.19	+0.81
C. Bahlman.....	11.13	11.25	11.50	+0.12	+0.37
C. G. Sutton.....	¹ 22.15	¹ 22.90	¹ 23.00	+0.75	+0.85
G. B. Taylor.....	16.88	15.25	18.50	-1.63	+1.62
E. W. Thornton.....	29.21	31.00	31.00	+0.79	+0.79
C. L. Black.....	¹ 16.80	¹ 16.35	¹ 16.88	-0.45	+0.08
	¹ 15.41	¹ 15.08	¹ 15.43	-0.33	+0.02
J. H. Bornmann.....	¹ 16.44	¹ 18.00	+1.56
	¹ 19.35	¹ 19.80	+0.45
W. C. Geagley.....	16.37	17.10	17.00	+0.73	+0.63

MISCELLANEOUS ICE CREAMS.

ANALYST	ROSE- GOTTLIEB	WENDLER	GRIGSBY	VARIATIONS FROM ROSE-GOTTLIEB	
		Sum of (a) and (b) readings	Sum of (a) and (b) readings	Wendler	Grigsby
H. B. Burnett.....	9.11	9.40	9.20	+0.29	+0.09
	6.04	5.60	5.80	-0.44	-0.24
R. E. Stevenson.....	9.00	8.84	9.15	-0.16	+0.15
	6.30	5.23	5.13	-1.07	-1.17
C. Bahlman.....	11.02	10.60	10.75	-0.42	-0.27
C. G. Sutton.....	¹ 11.63	¹ 11.45	¹ 11.75	-0.18	+0.12
G. B. Taylor.....	¹ 13.47	13.00	¹ 13.80	-0.47	+0.33
E. W. Thornton.....	14.77	14.80	14.66	+0.03	-0.11
C. L. Black.....	¹ 11.06	¹ 10.13	¹ 10.90	-0.93	-0.16
	¹ 11.03	¹ 9.83	¹ 10.93	-1.20	-0.10
J. H. Bornmann.....	¹ 9.63	¹ 9.73	+0.10
	10.29	¹ 9.98	-0.31
H. A. Halvorson.....	¹ 10.46	10.70	10.30	+0.24	-0.16
	¹ 12.28	12.40	12.60	+0.12	+0.32
W. C. Geagley.....	¹ 11.31	¹ 11.40	¹ 11.20	+0.09	-0.11

¹ Average of two or more results.

milk, and ice cream. The report of Mr. Keister as a collaborator during the past season shows a large amount of painstaking thorough work, and is so extensive and complete as to necessitate its incorporation herewith separately under Tables 3 and 4.

The following comments have been submitted by the collaborators:

COMMENTS BY COLLABORATORS.

C. L. Munroe, Miner Laboratories, Chicago, Ill.: Was unable to obtain any results by the Wendler method. In only one or two instances (while working with other samples of unsweetened condensed milk) was I able to obtain a fat column and those obtained were very unsatisfactory, as they were not clear and did not have a definite lower limit. In all other cases no fat column at all was obtained. I then tried several minor variations in the procedure outlined in the written directions, as follows: After mixing the sample, water, reagent, and isobutyl alcohol (this mixing was done very carefully and thoroughly in every case, shaking each bottle five minutes), I heated the bottles in a water bath at 55°C. for 20 minutes, with shaking. Then centrifuged at high speed for 5 minutes, added boiling water up to the neck, centrifuged 2 minutes at high speed, added boiling water to bring fat column up into neck, centrifuged 1 minute slowly and 1 minute at high speed. Obtained a fat column, but it was not clear, contained lumps of casein, and the lower limit was not sharply defined.

W. D. Bigelow, National Cannery Association, Washington, D. C.: When the samples were examined in this laboratory we had mislaid your letter, and therefore we examined them by the method that we ordinarily employ; that is, making only two extractions. It came to my attention afterwards that your letter directed three extractions. The results of Messrs. Fitzgerald, Miller, and Donk are, therefore, 0.02 or 0.03 lower than they would have been if your directions had been followed exactly.

F. C. Broeman, F. C. Broeman & Co., Cincinnati, O.: The Babcock method and its modifications gave such poor results on the sugared evaporated milk that I would not deem them worthy of consideration. Charring and indistinct separations made it impossible to get check readings on different determinations of same sample.

J. H. Bornmann, U. S. Food and Drug Inspection Laboratory, Chicago, Ill.: Brinsmaid method: While this method gives good results, it is objectionable because it requires the coating of the bottle with glycerol and heating in boiling water for 20 minutes.

Grigsby method: This method has the advantage of rapidity and simplicity over the Brinsmaid method. One disadvantage is the difficulty of knowing when just enough, and not too much, acid has been added.

Wendler method: Whereas this method gave satisfactory results on milk, it was a failure on all the other products. The fact that a comparatively harmless solution at moderately high temperature is used in place of sulphuric acid at a high temperature is one point in favor of this method. The fat column in the case of milk was very clear and sharply defined, which was not often the case to so marked a degree with the other methods employed.

The Roese-Gottlieb method requires a longer lapse of time before the results are obtained, but the total time spent on the manipulation is not so great when compared with the time spent on a centrifugal method that gives trouble. It is evident that the fat can be weighed more accurately than measured.

The Leach method (precipitation with copper sulphate and removal of the sugar solution in sweetened milk) seems to give low results.

H. A. Halverson, State Dairy and Food Department, St. Paul, Minn.: After a large amount of preliminary work I found that I was able to obtain very satisfactory results with the Wendler method on whole milk, evaporated milk, and ice cream; with the Brinsmaid method on evaporated milk; and with the Grigsby method on ice cream. The importance of sufficient preliminary experience with all these methods, especially the Wendler method, cannot be overestimated. The following paragraphs show briefly the results that can be obtained. All the results are averages of closely agreeing duplicates.

Wendler method: On three samples of miscellaneous whole milks, results (the average of (a) and (b) readings) on two samples ran 0.04 and 0.09% higher and results on one sample ran 0.01% lower than the corresponding results by the Roese-Gottlieb method. On four samples of miscellaneous evaporated milks, results (sum of (a) and (b) readings) on three samples ran 0.02, 0.03, and 0.01% higher and results on one sample ran 0.04% lower than the corresponding Roese-Gottlieb results. On six samples of miscellaneous ice creams, results (sum of (a) and (b) readings) on five samples ran 0.21, 0.12, 0.30, 0.04, and 0.24% higher and results on one sample ran 0.14% lower than the corresponding Roese-Gottlieb results.

Brinsmaid method: On four samples of miscellaneous evaporated milk, results (sum of (a) and (b) readings) ran 0.22 and 0.03% higher and results on the other two samples ran 0.09 and 0.09% lower than the corresponding Roese-Gottlieb results.

Grigsby method: On five samples of miscellaneous ice creams, results (sum of (a) and (b) readings) on four samples were 0.21, 0.32, 0.35, 0.26% higher and results on one sample were 0.01% lower than the corresponding results by the Roese-Gottlieb method.

Roese-Gottlieb method: I consider this method superior to any other when great accuracy is required. An experienced analyst, with care, can obtain results that agree very closely, say, within less than 0.1%. In order that different analysts may obtain concordant results on the same sample, it is more desirable (due to errors made in measuring) to weigh the sample directly rather than to take 10 cc. of solution containing 40 grams in 100 cc. This is especially true in the case of ice cream, which usually contains a large amount of air even after being melted. I think that results nearer the truth would be obtained if the directions required that the sample be weighted out directly.

E. W. Thornton, Department of Agriculture, Raleigh, N. C.: I experienced some difficulty in the Wendler method on ice creams, due apparently to slight saponification of the butterfat. The results reported on the ice cream by this method are on a pure ice cream which contained no thickening agent.

My results on evaporated milk are on the sample No. 1 (unsweetened) which was sent to me. Both centrifugal methods on this product gave very clear and satisfactory fat readings, but with a uniform difference of 0.2% between readings (a) and (b). I was unable to obtain satisfactory results by either the Wendler or Brinsmaid methods on sweetened condensed milk.

C. N. Austin, Sears, Roebuck & Co., Chicago, Ill.: Roese-Gottlieb method: In this work the fat flasks were dried to constant weight before the ether extracts were added. A counterpoise flask was used, which underwent the same heating as the flasks used in weighing the fat. It was found to be impossible to weigh a series of flasks with uniform rapidity. To compensate for the change in weight of the flasks, which takes place when they are removed from the desiccator, each one was allowed to stand on the balance pan until constant weight had been reached before a weighing was recorded. Another error that was difficult to avoid was caused by casein or water-soluble material passing through the filter with the ether extracts. To avoid this the extracts were allowed to stand about 30

minutes, and the filters were moistened with petroleum ether before adding the extracts. In making the extractions petroleum ether redistilled and coming over below 60° was used. Blanks were run to allow for the extractive matter in the reagents and nonvolatile residue in the ether. These caused a correction of 0.02 to 0.04 in the percentage. We were able to obtain results checking within 0.05%.

Babcock method: The modifications of the Babcock methods suggested were used in making determinations of the fat on the three samples submitted, as well as on a number of samples used for preliminary work. Bottles of the form specified could not be obtained in time for the work. The bottles used were of the ordinary form, reading to 10% and graduated to fifths of a per cent. The height from 0 to 8.0 on the stem of the 10% bottles was 1.5 cm. less than the height of the corresponding reading of the 8% bottles. A number of determinations were afterward made on other samples using the two kinds of bottles. This showed an average difference in the height of meniscus for the two kinds of bottles of only 0.01 on the scale. No correction was applied, however, since it was ascertained that the correction of -0.24, recommended for the Brinsmaid method, was originally determined from results obtained with bottles of the kind used by us. With all of the Babcock methods it was found difficult to obtain a sharply defined column of clear fat, due sometimes to a slight char, but more often to the presence of a slight amount of fine caseous material that prevented an accurate reading of the lower limit of the fat column. The fact that errors are multiplied by the necessity of using 9-gram samples would appear to be an added objection to the Babcock methods.

The Brinsmaid method was found to give results higher than the Roesse-Gottlieb results, even after the correction had been applied. However, the difference was not uniform for the three samples. The method gave results that check closely within themselves, and seemed to be as good for sweetened condensed milk as for evaporated milk.

The Grigsby method was found to be very unsatisfactory. It was very difficult to obtain fat columns free from curd or char, and even perfectly clear fat columns would sometimes give readings varying by as much as 0.2. The results were lower than those obtained by the other Babcock methods. With sweetened condensed milk this method was only applicable when the reagents were added to the drained curd. The presence of a small amount of water prevented the complete solution of the caseous material. When the results were tabulated the correction for the Brinsmaid method was applied to these results also as indicated in the instructions.

In the case of the Wendler method we substituted amyl alcohol for the isobutyl alcohol recommended, since the latter could not be obtained, and the former appeared to be effectual in preventing saponification of the fat. The method thus modified gave results with evaporated milk that accorded closely with those obtained by the Brinsmaid method, but with sweetened condensed milk no results could be obtained, since the precipitated curd did not dissolve completely.

H. S. Bailey, Bureau of Chemistry, Washington, D. C.: Just for my own satisfaction I tried to see how close duplicates could be obtained on a sweetened condensed milk, weighing up the original milk after dilution instead of merely using a 10 cc. pipette. Somewhat to my surprise the results were 8.996, 8.994, and 8.992. By using the weighing burette so that only one weighing more than the number of portions weighed out is required, very little more time is required than if we used a pipette, as there is no occasion for rinsing. To my mind the one drawback in the Roesse-Gottlieb method as outlined is the danger of using too much water in rinsing out the pipette. I find that if only 2 or 3 cc. are used there is no emulsion formed, while if one takes 10 cc. it is necessary to let the determination stand for a long time before it is possible to draw off the ether-fat column.

L. B. Burnett, Bureau of Chemistry, Washington, D. C.: Of the two centrifuge methods tried on whole milk, the Wendler method gave the best results, the fat column being perfectly clear, the lower meniscus horizontal. On evaporated milk the Wendler method did not work quite as well as on whole milk. More consistent results were obtained if the sample was not shaken during the heating, but rotated. With a little experience good results were obtained. On cream and ice cream the Wendler method seemed to give satisfactory results. The variations in the results on the second sample of cream and ice cream were not due to any fault of the method, as clear fat columns were obtained, but to the condition of the sample.

The Grigsby method was found to be satisfactory for sweetened condensed milk. The sample being diluted with one-third its weight of water and 12 instead of 9 grams being weighed out.

The Brinsmaid method did not appear to offer any special advantages, but satisfactory results were obtained with it on evaporated milk.

In the Wendler method in making up the fat column to the top of the graduations, it was found that if a dilute alcohol was employed instead of water the flocculent material was less apt to come to the top; however, this is not necessary if the sample is not shaken violently during the heating.

C. L. Black, U. S. Food and Drug Inspection Laboratory, Philadelphia, Pa.: I found the Wendler method most unsatisfactory in that in no case was I able to get all the curd dissolved by the amount of heating and shaking directed in the method, this being especially noticeable in the case of ice cream. The method would appear very unsatisfactory as a routine laboratory method in that, owing to the amount of vigorous shaking required, so few determinations can be made at one time, and these require so much more attention from the analyst than any of the ordinary methods used for similar products. In addition, it appears that the prolonged shaking seems to cause a decided loss of fat.

Geo. B. Taylor, State Board of Health, New Orleans, La.: Wendler method proved to be very unsatisfactory. In nearly every instance the clear fat would be underlain by curd. In a few instances the fat refused to rise.

It is my opinion that the Roesse-Gottlieb method as outlined (40 grams to 100 cc., 10 cc. = 4 grams taken) is unsatisfactory where materials heavy in fat are concerned. Small lumps of fat will invariably form when the solution is mixed, and remain in the pipette after the required amount is run into the Röhrig tube. When large amounts of fat are present it is practically impossible to get to constant weight by heating at the temperature of boiling water. An appreciable loss is almost always noted on heating for intervals of an hour.

During the last two years this laboratory has used, with good results, a slight modification of the Grigsby method for analyzing ice cream. We use 17.6 cc. of glacial acetic acid, mix well, but do not heat, adding commercial sulphuric acid to dark coffee brown color (about 10 cc.). This gives a clear reading. The results duplicate well.

On the other hand, it has been difficult to obtain good duplicates with the Roesse-Gottlieb method, especially with fruit ice creams. No method is satisfactory which requires the weighing out of 9 grams of sweetened condensed milk into an 8% Babcock milk bottle. This was done on triplicate samples, and worked by the Brinsmaid method, with no fat rising. A 40% solution was made up and 15 cc. equivalent to 6 grams were taken. No results were obtained. The Roesse-Gottlieb method is the only satisfactory method for sweetened condensed milk.

Clarence Bahlman, Board of Health, Cincinnati, Ohio.—When 18 grams of cream were treated with Wendler's reagent, there was a slight curd under the fat, when heated 5 minutes at 50°C. This was entirely avoided when the mixture of cream

and reagent was heated 10 minutes at 60°C. In the case of the evaporated milk, the same trouble was experienced when heating 5 minutes at 50°C., but was remedied by heating 10 minutes at 60°C. With the sweetened condensed milk, the Brinsmaid method gave considerable trouble. The fat layer showed a dark curd. Four tests were made; in only one were we able to obtain a satisfactory fat. Also, the Wendler method showed some curd when heating 5 minutes at 50°C., but gave clear separation when heated 10 minutes at 60°C.

J. T. Keister, Bureau of Chemistry, Washington, D. C.: In the Brinsmaid method very good readings were obtained in most cases and the results indicate that it gives figures varying only about 0.10 to 0.15 from the truth, when the readings are made to lower line of meniscus.

The Wendler method was a failure for evaporated milk on the 9-gram charge. Using a 6-gram charge was a considerable improvement, but as only two samples were tested by this modification no conclusion can be drawn.

The Grigsby method gave very satisfactory results on plain and nut ice cream and is about all that could be expected of a centrifugal method. In case of plain cream not a sufficient number of figures have been recorded to form any conclusion as to whether the correction -0.24 is necessary or not, as only two samples were examined, with some variation in results. In case of nut ice creams these results indicate clearly that the above correction is not necessary. The method worked successfully on the sample of pineapple cream, but in case of the other fruit creams (peach and strawberry) the method was not a success, the fat not being completely liberated and the fruit pulp also interfering with readings.

In case of the Wendler method, the results show that in its present form the method is not applicable to ice cream.

DISCUSSION OF RESULTS.

Reports from collaborators, most of whom reported determinations in duplicate, are on the whole a very satisfactory demonstration of the reliability of the Roese-Gottlieb method, especially when applied to such products as unsweetened and sweetened evaporated milk. With the exception of a few modifications, as suggested by some of the collaborators, the directions for carrying out the Roese-Gottlieb method will doubtless remain in the form already adopted as provisional. The preferred modification consists in weighing the sample. With this modification and one or two not very material changes in description, the method is in form for another season's study, anticipating its final adoption as official.

The associate referee selected, finally, for comparative study the methods devised by Wendler,¹ Brinsmaid,² and Grigsby.² The first of these, it is true, was described as applicable to the testing of milk, but preliminary trials of the method on other products led to the hope that good results might be forthcoming if the collaborators were directed to apply it to all samples submitted. The Brinsmaid method was devised for evaporated milk and the Grigsby method for ice cream. Unfortunately, in planning the work for the present year, the method described by Hunziker, and recommended for study at the meeting held in 1909, was overlooked.

¹ Z. öffent. Chem., 1906, 12: 41-58; Analyst, 31: 118.

² Correspondence from Mr. David Klein of the Illinois Food Commission.

There are, however, some results by this method reported by two of the collaborators, and these, together with information received from reliable sources, afford a basis for the belief that the method should be included in the coöperative study to be carried out next year. So far as can be judged from results reported by the collaborators, it appears that the methods devised by Brinsmaid, Grigsby, and Hunziker are worthy of further study, attention being given primarily to the products for which they were designed; and, in addition to these, there may probably be included the methods described by Beimling and Manchester. In the selection of methods it will, however, be borne in mind that a test like that described by Beimling has the serious defect arising from the probable solution of amyl alcohol in the fat column, thus causing too high results. A modified Roesse-Gottlieb procedure has for some time been under process of perfection in the dairy laboratory at the U. S. Bureau of Chemistry, but there is no assurance as yet that this is available for collaborative study with a view to adoption by this association. It may seem like a venture somewhat far from the aims of the association to put these modified centrifugal methods so much to the fore in our collaborative work in comparison with a method now well-nigh permanently established. There are, however, two purposes in view, either one of which seems to justify the past season's plan of work. In the first place, there is a persistent demand for a reliable quick method for factory control work or as a reliable sorting-out test for use in State and municipal food and dairy laboratories. In the second place, in the interest of sound policy and future stability in our work as analysts, this group of miscellaneous proposed so-called modified Babcock tests should be subjected to careful criticism and fair treatment. Then, any of these modifications that are not to be depended upon for the purposes for which they were devised should be consigned to oblivion. The supply of such methods in the last half dozen years has perhaps much exceeded the demand; most of them have gone into print without anything like adequate analytical backing; and the originators have been for the most part overconfident and somewhat lax in their descriptions. Some analysts, in fact, seem to imagine that one or two of these methods are applicable to all kinds of dairy products, from plain milk to ice cream and infant foods. The program which will be offered for the coming year will at any rate contemplate a weeding out among the score or more of these centrifugal methods with a view to determining which, if any, are really dependable for the products for which they were originally designed.

RECOMMENDATIONS.

It is recommended—

(1) That the Roesse-Gottlieb method be adopted as official for the determination of fat in milk and condensed milk, both unsweetened and sweetened.

(2) That a further study be made of the Roesse-Gottlieb method in the analysis of ice cream, milk powders, malted milks, and milk chocolates.

(3) That a special further study be made of modifications of the Babcock method as applied to condensed milk, both unsweetened and sweetened, and to ice cream.

TABLE 3.

Comparative study of fat methods for milk, cream, and evaporated milk.
MILK.

SAMPLE	WENDLER METHOD ¹				BABCOCK METHOD				ROESSE-GOTTLIEB METHOD
	(a)		(b)		(a)		(b)		
	Reading	Per cent fat	Reading	Per cent fat	Reading	Per cent fat	Reading	Per cent fat	
No. 1.....	3.95	3.95	3.75	3.75	3.75	3.75	3.55	3.55	3.69 3.696
No. 2.....	3.40	3.40	3.20	3.20	3.20	3.20	3.00	3.00	3.225 3.218

¹ Reading O. K.

CREAM.

METHOD	READING	PER CENT FAT	READING	PER CENT FAT	READING	PER CENT FAT
	(a)		(b)		(c) with glymol	
Wendler: ¹						
No. 1.....	17.9	17.88	17.30	17.29	17.40	17.39
No. 2.....	16.4	16.12	15.8	15.58	15.7	15.48
Babcock:						
No. 1.....	17.9	17.74	17.3	17.15	17.4	17.25
No. 2.....	15.9	15.70	15.3	15.11	15.4	15.20

¹ Found it difficult to get uniform readings with cream. Method not considered as reliable as Babcock.

Roesse-Gottlieb:

	Per cent fat
No. 1.....	17.32
No. 2.....	{ 15.16 15.23

TABLE 3.—Continued.
EVAPORATED MILK.

SAMPLE	WEIGHT OF SAMPLE	READINGS		PER CENT FAT				REMARKS
		(a)	(b)	¹ (a)×2	(b)×2	(a)×2 − 0.24	(b)×2 + 0.15	
BRINSMAID METHOD.								
a	grams							
	9.0947	3.8	3.6	7.52	7.12	7.28	7.27	Readings O. K.
b	9.0720	3.8	3.6	7.54	7.14	7.30	7.29	Slight undissolved curd.
	9.0622	3.8	3.6	7.54	7.15	7.30	7.30	Some charring.
c	9.0600	3.9	3.7	7.74	7.35	7.50	7.50	Do.
	9.0747	3.9	3.7	7.73	7.33	7.49	7.48	Reading O. K.
d	9.0550	3.9	3.7	7.75	7.35	7.51	7.50	Little brown "mud."
	9.0395	3.9	3.7	7.76	7.36	7.52	7.51	Cloudy fat but good read- ing.
e	9.9780	3.9	3.7	7.73	7.33	7.49	7.48	Do.
	9.1914	4.4	4.2	8.61	8.22	8.37	7.91	Very good readings.
f	8.9388	4.3	4.1	8.66	8.25	8.42	8.02	Do.
	9.0417	4.10	3.90	8.16	7.76	7.92	7.91	Readings O. K.
g	9.0276	4.15	3.95	8.26	7.87	8.02	8.02	This one charred.
	9.0305	4.40	4.20	8.77	8.37	8.53	8.52	Slight char; not perfect.
h	9.1270	4.45	4.25	8.776	8.38	8.54	8.53	Do.
	9.0245	4.3	4.1	8.57	8.17	8.33	8.32	Some char.
	9.1210	4.2	4.0	8.29	7.89	8.05	8.04	Reading O. K.
WENDLER METHOD.								
a	9.0437	3.45	3.25	6.86	6.46	6.62	6.61	Undissolved casein, not a success.
	9.0722	3.30	3.10	6.54	6.15	6.30	6.30	
b	9.1882	3.55	3.35	6.95	6.56	6.71	6.71	Some white curd.
	9.0677	3.50	3.30	6.94	6.55	6.70	6.70	Do.
c	9.1330	3.20	3.00	6.37	5.98	6.13	6.13	Much white curd.
	9.0575	3.10	2.90	6.18	5.77	5.94	5.92	Do.
i	6.0700	2.60	2.40	7.71	7.12	7.47	7.27	Clear reading; (a) × 3.
	6.2470	2.70	2.50	7.77	7.20	7.53	7.35	Do.
j	6.0235	2.50	2.30	7.48	6.87	7.24	7.04	Reading O. K.; (a) × 3.
	6.0651	2.50	2.30	7.42	6.82	7.18	6.97	Slight brown "mud."
ROMER-GOTTLIEB METHOD.								
SAMPLE	PER CENT	SAMPLE	PER CENT	SAMPLE	PER CENT	SAMPLE	PER CENT	
a.... {	7.172	c.... {	7.41	e.... {	8.172	h.... {	8.05	
	7.144		7.41		8.197		8.06	
b.... {	7.10	d.... {	7.478	f.... {	7.765	i.....	7.12	
	7.15		7.519		7.779	7.457		
				g.....	8.59			

¹ (a) is corrected to 9 grams.

TABLE 4.
Comparative study of fat methods with ice cream.

GRIGSBY METHOD.

SAMPLE	VARIETY	WEIGHT OF SAMPLE FLM. (GRAMS)	READINGS		PER CENT FAT					REMARKS
			(a)	(b)	(a) + (b) ¹	(a) × 2	(b) × 2	(a) × 2 - 0.24	(b) × 2 + 0.16	
a	Vanilla.....	9.0915	4.95	4.78	9.62	9.80	9.40	9.56	9.55	Reading O. K.
		9.2712	5.10	4.90	9.70	9.90	9.51	9.66	9.66	Do.
		9.0716	4.55	4.35	8.82	9.023	9.60	8.78	8.76	Reading perfect.
b	Chocolate.....	9.1961	4.60	4.40	8.82	9.04	8.62	8.80	8.77	Few small lumps of chocolate in neck; not objectionable.
		9.0120	4.60	4.40	8.97	9.18	8.78	8.94	8.93	Reading O. K.
		9.0232	4.70	4.50	9.16	9.37	8.97	9.13	9.12	Do.
c	Vanilla.....	9.0484	4.05	3.85	7.86	8.06	7.66	7.82	7.81	Fruit pulp was in neck of bottle, but separated from fat, and did not interfere with readings.
		9.0067	4.10	3.90	7.98	8.19	7.77	7.95	7.93	Do.
		9.2447	3.50	3.30	6.61	6.81	6.42	6.57	6.57	Fat and pulp not in contact; no interference with reading.
d	Pineapple.....	9.1150	3.50	3.30	6.71	6.91	6.50	6.67	6.65	Fairly good reading; pulp interfered somewhat.
		9.0820	4.00	3.80	7.73	7.93	7.52	7.69	7.67	Only fair reading, pulp interfered.
		9.1520	4.20	4.00	8.06	8.26	7.86	8.02	8.01	Do.
e	Peach.....	9.5625	4.70	4.50	8.65	8.84	8.46	8.60	8.61	Reading O. K.; nuts in neck of bottle, but separated from fat.
		9.0225	4.40	4.20	8.58	8.78	8.38	8.54	8.53	Very good reading obtained by heating H ₂ O bath slightly above 80°, which caused separation of nuts from fat.
		9.0210	3.90	3.70	7.58	7.78	7.38	7.54	7.53	Fruit pulp interfered with readings, some fat being entangled with pulp.
f	Strawberry.....	9.2230	3.8	3.60	7.27	7.52	7.02	7.78	7.17	Do.
g	Maple nut.....									
h	Peach.....									

WENDLER METHOD.

a	Vanilla.....	9.1160	4.30	4.10	8.28	8.49	8.08	8.25	8.23	Only fair test some curd. Too much curd to read accurately.
		9.0710	4.5	4.30	8.72	8.92	8.52	8.68	8.67	
		9.0855	4.45	4.25	8.61	8.81	8.42	8.57	8.57	
b	Chocolate.....	9.1282	4.50	4.30	8.68	8.87	8.48	8.63	8.63	White "mud" at bottom of fat column; not a success.
		9.1370	4.60	4.40	8.86	9.06	8.66	8.82	8.81	Do
		9.0612	4.50	4.30	8.74	8.94	8.54	8.70	8.69	White "mud" reading only approximate.
c	Vanilla.....	9.1090	4.00	3.80	7.70	7.90	7.66	Do.
		9.0730	4.00	3.80	7.74	7.93	7.69	White curd or "mud" at bottom of fat column.
		9.0690	3.25	3.05	6.44	Do.
d	Pineapple.....	9.1540	3.25	3.05	6.45	White curd or "mud;" not a success.
										Do.
e	Peach.....									

ROBER-GOTTLINE METHOD.

SAMPLE	VARIETY	PER CENT FAT	SAMPLE	VARIETY	PER CENT FAT
a	Vanilla.....	9.58	e	Peach.....	7.62
b	Chocolate.....	9.57	f	Strawberry.....	7.66
c	Vanilla.....	9.02	g	Maple nut.....	8.136
d	Pineapple.....	9.03	h	Peach.....	8.128
		9.216			8.75
		9.23			8.776
		8.14			8.47
		8.16			8.508

¹ Corrected to 18 grams.

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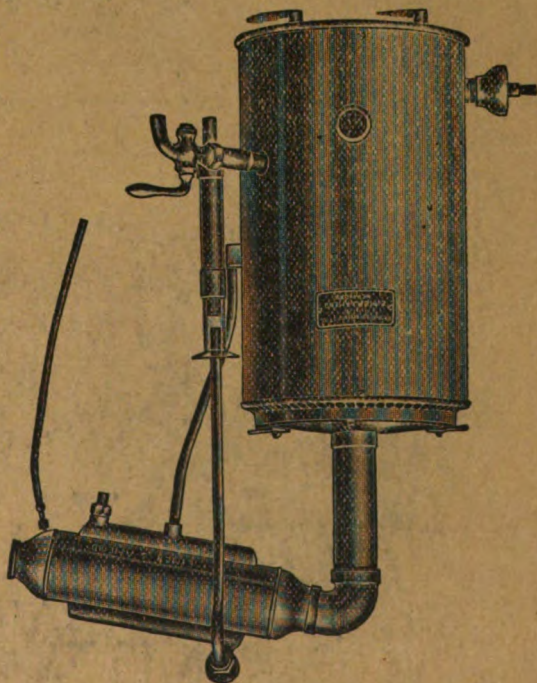
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